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Demonstration of Entrained Solids and Sr/TRU Removal Processes with Archived AN-107 Waste

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Prepared for BNFL, Inc.
under contract W375-98-LC-4168

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SUMMARY

Waste from Hanford underground storage Tank 241-AN-107 is a candidate low-activity waste (LAW) for Envelope C. Envelope C wastes require pretreatment to remove entrained solids, radioactive strontium, transuranics, radioactive cesium, and technetium before immobilization. The initial baseline pretreatment process includes plans for entrained solids removal by crossflow filtration, Sr/TRU precipitation with added strontium and iron, and Sr/TRU precipitate removal by crossflow filtration. However, studies have shown that entrained solids and the Sr/Fe precipitates were very difficult to filter from candidate Envelope C waste. An alternative pretreatment process being developed uses permanganate instead of iron. Permanganate treatment has been shown to be effective for decontaminating waste from Hanford Tank SY-101.

Small-scale experiments with archived AN-107 waste were conducted by Battelle to determine the effectiveness of the permanganate treatment process. These tests were conducted in three rounds of experiments. The early tests showed that permanganate treatment alone would provide adequate TRU removal, however, it would not provide adequate Sr removal. The second set of experiments showed the preferred Sr/TRU removal process involved addition of strontium and permanganate. Because the composition of the archived waste had been altered by past pretreatment tests (diluted and removal of cesium and settled solids), the final set of experiments included tests with actual AN-107 diluted feed. These tests identified conditions that should provide adequate Sr/TRU decontamination.

The work reported here was conducted to evaluate process conditions with two, 1-L batches of archived AN-107, which provided a volume of waste large enough for crossflow filtration studies. One, 1-L sample of archived waste was adjusted to 1M hydroxide and used for entrained solids removal tests. Following this test, Sr/TRU removal was accomplished by addition of a strontium nitrate solution followed by sodium permanganate solution. The resulting precipitate was used for crossflow filtration tests to demonstrate the performance of a 0.1-um filter element.

Even though many of the entrained solids were removed from the archived AN-107 sample by previous pretreatment testing (settle/decant and deep bed filtration from the ion exchange column), crossflow filtration was found to be impractical to remove the remaining solids due to very low filtrate flux rates (0.0079 gpm/ft^2). Therefore, AN-107 waste was treated for Sr/TRU removal with the entrained solids present. The entrained solids were then removed along with the Sr/TRU precipitate, in a single filtration step. Crossflow filtration tests were conducted in the Cell Unit Filter (CUF) system with the Sr/TRU precipitated waste. The filterability of archived AN-107, as determined by filter flux rate, increased by an order of magnitude after the pretreatment process (average of 0.11 gpm/ft^2). The pretreated waste could be effectively filtered by crossflow filtration.

Results were obtained from experiments with archived AN-107 samples treated at two different target reagent concentrations, 1M hydroxide, 0.075M strontium, and 0.05M permanganate, and 0.8M hydroxide, 0.05M strontium, and 0.03M permanganate. Approximately 1-L of archived AN-107 were treated in each experiment. Decontamination of strontium-90 and TRU (primarily

Am-241) in the supernatant was greater than needed to meet the immobilized low-activity waste (ILAW) requirements (less than 100 nCi/g TRU and less than 20 Ci/m^3 Sr-90 in the final ILAW). The target DFs were 10 for Sr-90 and 5 for Am-241. The stronium-90 decontamination factors (DFs) obtained were consistently greater than 20 and the Am-241 DF was 10 and greater. Removal of Eu isotopes 154 and 155 was slightly less than the Am DFs. These DFs include the contribution from the removal of the entrained solids although this was relatively small. The removal of the entrained solids accounted for very little Sr-90 removal and about 10% of the DF for Am-241 because the archived waste samples had most of the solids already removed.

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1.0 INTRODUCTION

BNFL Inc. was awarded the Privatization Contract for treatment of Hanford underground storage tank wastes as part of the River Protection Project-Waste Treatment Plant (RPP-WTP). In Part B-1, Battelle is conducting technology development and demonstration of process flowsheet steps for BNFL. Entrained solids removal by crossflow filtration is the first proposed process step in pretreatment. Filtration should remove sufficient solids to prevent plugging of the ion exchange columns downstream and to ensure that insoluble radioactive strontium and transuranic isotopes (TRU) are removed. These solids are then to be concentrated and returned to the U.S. Department of Energy (DOE). The RPP-WTP Privatization Contract (2000) specifies certain isotopic, chemical, and physical limits for the entrained solids returning to the DOE double-shell tanks.

Three candidate low-activity waste types have been identified: Envelope A, Envelope B, and Envelope C. Treatment and disposal of the liquid (supernatant) fraction of Envelope C wastes, such as Tank 241-AN-107, requires additional treatment to remove transuranics and radioactive strontium. Because of the high concentration of organic complexants in this waste (Complexant Concentrate waste), conventional separation processes (e.g., ion exchange) are not effective.

During Part A-1 of privatization, Savannah River Technical Center (SRTC) developed a Sr/TRU removal process involving isotopic dilution and precipitation with added strontium and iron (SRTC 1997a, 1997b, 1997c, and 1997d). While this treatment process provided the necessary supernatant decontamination, the resulting precipitate could not be filtered. The search began for an alternate treatment process. Battelle proposed permanganate be examined as an alternative, because it had been demonstrated to work with waste from Hanford Tank SY-101, which also contained high levels of organic complexants (Orth et al. 1995).

Permanganate has been examined as an oxidant for complexing agents (Orth et al. 1995), solubilizing chromium (Rapko et al. 1995, Rapko 1998), and oxidation of technetium species to pertechnetate (Schroeder et. al 1998) in tank wastes. Permanganate was found to oxidize chromium first, then organic carbon, and lastly nitrite. For wastes such as Tank SY-101, the chromium in the sludge consumes as much as half the permanganate. Orth et al. recommended permanganate doses of 0.1M for decomplexing SY-101 type wastes. At this level of permanganate, decontamination factors (DF) of > 143 were obtained for Sr and 28.5 for Pu. AN-107 does not have the high chromium values in the sludge so permanganate is expected to be effective at lower concentrations.

Permanganate is also used as a precursor to MnO_2 and/or $Mn(OH)_2$ coprecipitants via the "Method of Appearing Reagents" (Krot et al. 1996). The method of appearing reagents requires the addition of a reductant to the waste to be treated. However, for Hanford wastes this is not necessary because reductants are already present in the waste. The resulting solids are effective coprecipitants for Pu and other TRU elements but generally not as effective as iron precipitates. Decontamination factors of greater 100 have been reported for various simulated waste streams.

The objective of this work was to demonstrate the entrained solids and Sr/TRU removal processes with archived AN-107 waste before proceeding with the integrated processing of AN-107 diluted feed. Similar to entrained solids removal tests for AW-101 (Brooks et. al 1999), tests

were planned to determine the permeability of an Envelope C feed through a single element filter as a function of transmembrane pressure, axial velocity, solids concentration, and time. The archived AN-107 waste was used to demonstrate the treatment scheme for Sr/TRU removal developed from small-scale tests conducted at Battelle with waste simulants and actual waste involving strontium and permanganate addition. Supernatant decontamination data were obtained from two treatment levels; 0.075M Sr and 0.05M permanganate, and 0.05M Sr and 0.03M permanganate. Crossflow filtration tests were conducted with waste treated with 0.075M Sr and 0.05M permanganate to determine the efficiency for Sr/TRU solids removal. In addition, the efficiency of back pulse and chemical cleaning on the filter performance was evaluated. The chemical and radiochemical composition of the supernatant and filtrates were measured to determine efficiency of the Sr/TRU removal process.

This report contains the results of entrained solids removal, Sr/TRU decontamination, and Sr/TRU solids removal testing conducted at Battelle with archived AN-107 waste. Test conditions and experimental procedures are described in Section 2.0. Results from entrained solids removal and treatment with added Sr and permanganate are described in Section 3.0. The major conclusion and recommendations that evolved from this work are given in Section 4.0. The appendices contain the test instruction, data sheets, logbook entries, analytical data, calculation, and staff role/responsibilities for this work.

2.0 TEST CONDITIONS AND EXPERIMENTAL PROCEDURES

The conditions for conducting the entrained solids and Sr/TRU removal tests were detailed in Sr/TRU Precipitation and Ultrafiltration Test Specification (Townson 1998) issued by BNFL. The Test Specification was used to prepare a Test Plan (TP 29953-013) that described the general requirements for the Sr/TRU removal tests to be conducted at Battelle. The actual test was conducted in accordance with Test Instruction-29953-041, which was specific to the Sr/TRU Removal test described in this report for archived AN-107. Deviations from the test instructions were necessary. The additional Sr/TRU precipitation experiment was conducted in accordance with Test Instruction-29953-063.

2.1 Description of Archived AN-107 Sample

The archived AN-107 material used for this test was collected, diluted, settled solids removed, and cesium ion exchanged prior to its use for the BNFL project (Hendrickson 1997). It was collected as 45 grab samples in 125-mL bottles taken during January 1997. Approximately 5.4 liters of tank waste was then transferred to 222-S laboratory and 0.53M sodium hydroxide was added to dilute the waste to 5M sodium and to a free hydroxide concentration of 0.24M. The supernatant was not filtered prior to cesium ion exchange. Instead, the solids were allowed to settle and the supernatant was decanted and sent through the crystalline silicotitanate loaded columns. Analysis of the waste after cesium removal indicated the free hydroxide to be 0.126M. Following cesium removal the sample was transferred to PNNL in five 1-L poly bottles where it has been stored in the Shielded Analytical Laboratory (SAL) hot cells in the Radiochemical Processing Laboratory (RPL).

In June 1999, one liter of this archive AN-107 (1242.62 g) was adjusted with NaOH pellets to achieve a target concentration of 1M free hydroxide. This material was then transferred to the High-Level Radiochemistry Facility (HLRF) hot cells and placed into the CUF system for entrained solids removal testing.

2.2 Crossflow Filtration

The River Protection Project Waste Treatment Plant (RPP-WTP) (1996) flowsheet uses cross-flow filtration as the solid/liquid separation technique. Unlike traditional dead-end filtration, which has a declining filtration rate caused by the growth of a filter cake on the surface of the filter medium, in cross-flow filtration, the filter cake is swept away by the fluid flowing across it. This filtration method is especially beneficial when there are very fine particles and when system simplicity is required.

One of the applications of crossflow filtration is to remove the entrained solids from the waste. The filtration should remove sufficient solids to prevent plugging of the ion exchange column downstream and to ensure that insoluble Sr-90 and transuranic isotopes are removed. Another application of crossflow filtration for Envelope C wastes is to remove the Sr/TRU precipitate from the treated supernatant. The proposed flowsheet for Envelope C waste shows two sequential solids removal steps: First removal of the entrained solids, then Sr/TRU solids removal after Sr and permanganate treatment.

Crossflow filtration tests were conducted in the HLRF hot cells with the Cell Unit Filter (CUF) system. The CUF had the following specifications:

- Mott sintered stainless steel filter, 0.1 micron rating, 24 in. long and 3/8 in. internal diameter (total area 0.196 ft^2)
- Re-circulation flow with a maximum linear crossflow velocity of 16.4 ft/s along the axis of the filter
- Maximum transmembrane pressures of 80 psi
- Temperature control of 25 $\pm$ 5°C during operation.

The system was fabricated based on modifications of the CUF system designed by SRTC. It is described in detail in Brooks et al. (1999). Unlike that used for the AW-101 testing, the filter used in this work was a Mott 0.1 μm-rated filter designed specifically for liquid service. Conditions for filtration tests were specified in Test Instruction 29953-041, Appendix A.

2.3 Sr/TRU Removal Conditions

Supernatant from Envelope C waste contains Sr-90 and TRU levels that are too high to meet immobilized low-activity waste (ILAW) requirements. The BNFL targets for ILAW are less than 100 nCi/g TRU and less than 20 Ci/m^3 Sr-90 in the final ILAW. For AN-107 waste, this translates to target decontamination factors (DF) of approximately 10 for strontium (90% removal) and 5 for TRU (80% removal). Since over 90% of the TRU in AN-107 is due to Am-241, a decontamination factor of 5 was established for Am-241.

Experimental conditions for Sr/TRU removal were determined based on results from small-scale batch experiments with archived AN-107 waste (Hallen et al. 2000). The hydroxide concentration was increased by the addition of sodium hydroxide as solid pellets or 19M (50 wt%) solution. Strontium nitrate and sodium permanganate were added as 1M solutions. The results from the small-scale experiments suggested that adequate Sr/TRU removal could be obtained at a hydroxide concentration as low as 0.5M and reagent concentrations as low as 0.05M strontium and 0.03M permanganate for the archived AN-107 sample. But conservative conditions were chosen for the filtration tests, 1M hydroxide and reagent concentrations of 0.075M for strontium and 0.05M for permanganate, because the AN-107 diluted feed was more concentrated and contained more entrained solids than the archived waste. As part of an effort to prepare feed for sulfate removal tests, a later Sr/TRU removal experiment was conducted with archived AN-107 adjusted to 0.8M hydroxide and reagent concentrations of 0.05M strontium and 0.03M permanganate. This later sample was not tested in the CUF. Target compositions and volumes are shown in Table 2.1 for both experiments

Table 2.1. Sr/TRU Target Concentrations and Volume for the Two Experiments Conducted with Archived AN-107

High Conc.	Target Concentration (M)	Target Volume (L)	Low Conc.	Target Concentration (M)	Target Volume (L)
initial waste	-	0.82	initial waste	-	1.00
NaOH	1	0.88	NaOH	0.8	1.05
$Sr(NO_3)_2$	0.075	0.95	$Sr(NO_3)_2$	0.05	1.11
$NaMnO_4$	0.05	1.00	$NaMnO_4$	0.03	1.14

2.4 Experimental

All Sr/TRU and solids removal tests were performed in shielded process cells located in the RPL at Hanford. The CUF system, located in the HLRF, was used for entrained solids and Sr/TRU precipitate removal tests. Test Instruction 29953-041, Appendix A, was used to conduct the entrained solids removal test, Sr/TRU removal at high reagent concentration, and Sr/TRU solids removal test. These tests were conducted from July 16 to July 28, 1999. The flowsheet describing these tests is shown in Figure 2.1.

Efforts were made prior to beginning this testing to ensure that clean water fluxes equal to or greater than the original factory specification were achieved. To do this, the pump and filter were changed out and remaining particulates were removed from the CUF by recirculating a side-stream through a 0.05 μm-rated cartridge filter. The clean water flux was then measured over the course of one hour. During this time, the average filtrate flux was 0.98 gpm/ft^2 at 20-psi pressure differential and an axial velocity of 11.6 ft/s. The clean water flux did not decline during this time. Following the hour long testing at 20 psid, the filter was tested at 10 and 30 psid for 20 minutes each. The filtrate flux remained relatively constant at 0.395 and 1.41 gpm/ft^2, respectively. These values were above the Mott reported values of 0.37 gpm/ft^2 for 10 psid and 0.72 gpm/ft^2 for 20 psid. The deionized water was drained from the system. It is estimated that approximately 100 mL of deionized water remained in the CUF after draining. This residual water diluted the archived AN-107 sample by roughly 10%.

The entrained solids were tested in the CUF under two conditions rather than the original 6 point matrix due to low filtrate fluxes. If the filtrate flow were less than 10 mL/min during the test, BNFL had specified that the test should be discontinued, the filter should be backpulsed, and a new condition should be tested. By stopping the test prematurely, unnecessary data would not be taken and further filter fouling would be prevented. The test conditions studied are shown in Table 2.2. When the flux was found to be very low for the first condition, two backpulses were attempted. Because of the low filtrate flux, only a small quantity of material could be collected in the backpulse chamber and two short backpulses were performed. A higher pressure was then tested to see if an increased flux were possible. Once again, the filtrate flux was excessively low and the test was discontinued. The entrained solids and filtrate were drained from the CUF and placed back into the original container. Samples were taken during the entrained solids removal test but were not analyzed.

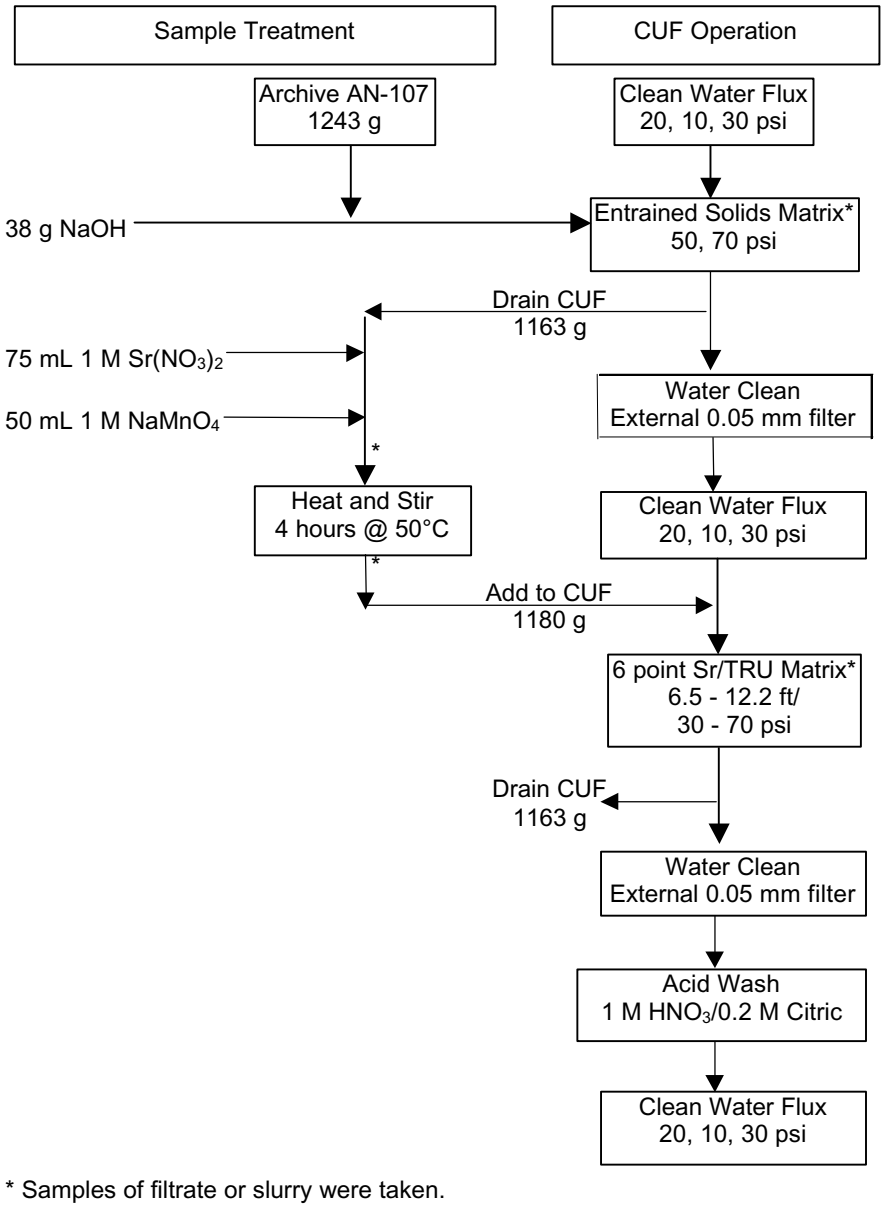

Figure 2.1. Flow Chart of Archive AN-107

Table 2.2. Test Conditions Studied for Entrained Solids Removal from Archived AN-107

Condition	Crossflow Velocity (ft/s)	Transmembrane Pressure (psi)
1	12.2	55
3	9.3	70

The CUF was drained, rinsed once with 0.2M NaOH, and then rinsed and the filter backpulsed multiple times with DI water to achieve a neutral pH. Once neutral conditions were reached, a 0.05 μm cartridge filter was attached to the CUF and the remaining particulate matter was removed. Clean water fluxes were measured and found that the original fluxes had been recovered. The system was drained in preparation for filtration of the Sr/TRU precipitate. Once again approximately 100 mL of DI water left in the CUF further diluted the archived AN-107 during the Sr/TRU precipitation filtration test.

The archived AN-107 sample was transferred to an Erlinmeyer flask and placed on a stir plate. The sample was not heated to 50°C before reagent addition as originally specified in the Test Specification. Small-scale test had shown higher DFs when the reagents were added at ambient temperature, then digested after the addition was complete. At ambient cell temperature, approximately 75 mL of a 1M solution of $Sr(NO_3)_2$ was added to the archived AN-107 sample over the course of 5 minutes. The original solution was dark brown, but during addition, white precipitate could be seen forming. Approximately 30 minutes later 50 mL of a 1M solution of $NaMnO_4$ was added to the archived AN-107 sample over the course of 4 minutes. These quantities were added to produce target concentrations of 0.075M Sr and 0.05M permanganate in the final treated sample. The slurry was then heated to 50°C with constant stirring and remained at temperature for 4 hours. The slurry was allowed to cool overnight and then it was added to the CUF for filtration testing.

Six conditions were tested with the Sr/TRU precipitated, archived AN-107 sample in the CUF. The conditions are shown in Table 2.3. It was not possible to maintain the target flows at the required pressures. This may be due to the higher viscosity associated with the Sr/TRU precipitate or pump stator wear.

Table 2.3. Test Conditions Studied With Archive AN-107 Sr/TRU Solids Removal

Condition	Crossflow Velocity (ft/s)	Transmembrane Pressure (psi)
1	11.2	53
2	11.8	38
3	8.1	70
4	9.1	55
5	6.4	54
6	10.0	54

During the course of testing, samples of the slurry and filtrate were taken for chemical and radiochemical analysis. Slurry samples were taken before $Sr(NO_3)_2$ and $NaMnO_4$ addition and after the 4 hour heating and subsequent cooling of the Sr/TRU precipitate. Filtrate samples were taken during each of the 6 conditions. Two slurry samples were also taken after Condition 3 and at the end of the CUF testing. Not all samples taken were analyzed. A filtrate composite sample, MN-32, was made of filtrate samples taken during each test condition and analyzed. The sample identification and analyses performed on each sample are shown in Table 2.4.

Table 2.4. Samples and Their Required Analyses

Sample Description	Sample ID Number	Sample Type	Sample Preparation	Analytes
Waste after entrained solids test (initial waste)	MN-21	Slurry	0.45 um filter, acid digest	Sr-90, Am-241, Na, OH^-
Waste after Sr/TRU Removal Treatment	MN-22	Slurry	0.45 um filter, acid digest	Sr-90, Am-241, Na, OH^-
1st CUF Permeate Sr/TRU Solids Removal	MN-23	Filtrate	acid digest	Sr-90, Am-241, Na
Final CUF Slurry	MN-28	Slurry	acid digest	Sr-90, Am-241, Na
Final CUF Permeate	MN-31	Filtrate	acid digest	Sr-90, Am-241, Na
Composite CUF Permeate	MN-32	Filtrate	acid digest	Sr-90, Am-241, Na

Test Instruction 29953-063 (see Appendix B) was used to conduct the Sr/TRU removal experiment at low reagent concentration. This experiment was conducted on October 26, 1999, to prepare Sr/TRU treated waste for sulfate removal scoping tests. Starting with 1-L of archived AN-107 waste, 48 mL of 50 wt% (19M) NaOH was added to give a calculated free hydroxide concentration of 0.8M. A 50-mL sample of caustic adjusted waste was removed for analyses (MR-01 and MR-02) and density determination. Then at ambient cell temperature, 57 mL of 1M $Sr(NO_3)_2$ was added with stirring over 6 min. The solution was stirred for an additional 9 min before adding 34 mL of 1M $NaMnO_4$ over 6 min. The waste was stirred for an additional 30 min before heating to 50°C and held there for 4 hours. After cooling to ambient temperature, the treated waste was filtered through a 0.45 um dead-end filter. The density of the filtrate was determined and a sample collected for analyses (MR-03). Table 2.5 listed the samples and analyses required.

Table 2.5. Samples and Their Required Analyses

Process Variable	Sample ID Number	Sample Type	Sample Preparation	Analytes
Archived AN-107	MR-01	Slurry	acid digest	Sr-90, Am-241, Na
Archived AN-107	MR-02	Slurry	0.45 um filter, acid digest	Sr-90, Am-241, Na
Treated AN-107	MR-03	Filtrate	acid digest	Sr-90, Am-241, Na

2.5 Chemical Analyses

All of the chemical analyses were conducted at Battelle. BNFL designated the analytes of interest and minimum reportable quantity in the test specification (see test instructions in appendix). Because the archived AN-107 sample had most of the radioactive cesium removed, Am-241 concentration could be determined directly by gamma energy analysis along with the Eu isotopes 154 and 155. Relatively high levels of Cs-137 raise the gamma background level in the detector due to Compton scattering, thereby making it difficult to detect other, lower-level gamma emitters, especially those having gamma energies below that of Cs-137. The Sr-90 concentration was determined by chemical separation followed by beta counting. Sodium concentration was determined by inductively couple plasma-atomic emission spectrometry, as well as the other metals listed in the test specification

The samples taken during tests in HLRF were transferred to the SAL for analytical sample preparation. All of the analytical results are included in Appendix C.

3.0 RESULTS AND DISCUSSION

The results of the testing and analyses are discussed below for entrained solids removal, Sr/TRU decontamination, and Sr/TRU solids removal from archived AN-107 samples.

The experimental and test conditions were defined by BNFL in Test Specification (Townson 1998, 1999) documents, change request documents, and direct communications with BNFL staff. General test plans were prepared for LAW crossflow filtration and Sr/TRU removal tests (TP-29953-004 and-013). Test instructions were prepared which detailed the specifics for conducting and documented deviations from the test specification for conducting tests with archived AN-107 waste. The test instructions were used to record the specific details of the tests, and are attached in Appendix A and B.

3.1 Entrained Solids Removal

The proposed pretreatment flowsheet shows entrained solids are removed from the double-shell tank wastes as a first step pretreatment. The entrained solids removal test was conducted with a caustic adjusted (1M hydroxide), archived AN-107 waste sample. The test demonstrated that the entrained solids present in this waste could not easily be removed by crossflow filtration. For entrained solids removal, the initial flux dropped in less than a minute to 0.023 gpm/ft^2 and within 5 minutes had dropped to 0.0074 gpm/ft^2 at 55 psi transmembrane pressure (TMP) and 12.2 ft/s crossflow velocity. To prevent further plugging of the filter, no further testing was conducted at this condition. An attempt was made to collect sufficient filtrate to backpulse (clean) the filter. Only a small quantity of material could be collected in the backpulse chamber and two short backpulses were performed. A second condition was then tested at 70 psi and 9.3 ft/s (Condition 3 of the test matrix). In this case, after 1 min the filtrate flux was 0.0079 gpm/ft^2. Testing was stopped at this point and entrained solids removal was determined to be not feasible for AN-107 waste.

The archived AN-107 sample had most of the entrained solids removed by settle/decant and deep bed filtration as part of the ion exchange column testing. No visible solids remained, yet the sample could not be easily filtered. This suggests fine particles or colloidal solids are suspended in the AN-107 sample that plug or foul the filter media. The high axial velocity of liquid across the filter surface did not help filter performance, which suggests that filter cake build up is not an issue. Entrained solids removal is expected to be worse for AN-107 diluted feed because it has approximately 1% settled solids and has not been diluted and used for ion exchange testing.

The archived AN-107 sample was drained from the CUF and Sr/TRU precipitation test was conducted on the sample containing the entrained solids. The samples collected during the attempted filtration of the entrained solids were not analyzed.

3.2 Sr/TRU Decontamination

Sr/TRU removal tests were conducted at two different target concentrations of caustic and reagent addition. To prepare the waste for the filtration tests using the CUF, chemicals were added to give a calculated final concentration of 1M hydroxide, 0.075M Sr, and 0.05M permanganate. Later as part of the sulfate removal scoping tests, Sr/TRU removal was determined for lower concentrations of added chemicals, 0.8M hydroxide, 0.05M Sr, and 0.03M permanganate. The results from these two experiments will determine the potential to minimize the addition of chemicals and the resulting amount of Sr/TRU removal solids for disposal as high activity waste.

Multiple samples were taken during the Sr/TRU removal tests and analyzed to determine the change in waste composition upon treatment. Samples were taken after various stages of treatment and filtration. The radionuclide composition of the treated samples was compared with the initial composition to determine the extent of decontamination. The initial waste composition is the composition after caustic addition and any dilution that may have occurred. The Decontamination Factor (DF) is defined as the concentration of the component in the initial waste divided by the concentration after treatment, corrected by the amount of dilution that occurred:

$$DF = [A]_i/([A]*MD)$$

where $[A]_i$ is the concentration of component A per mass in the initial sample, [A] is the concentration of component A per mass in the treated sample, and MD is the mass dilution, final mass of treated solution divided by the initial mass of solution. The final mass is determined by summing up the mass of initial waste and all dilution, adjustments, and/or reagent additions.

The archived AN-107 sample from the entrained solids test was used for the higher reagent concentration, Sr/TRU removal test. At ambient hot-cell temperature, approximately 75 mL of 1M$Sr(NO_3)_2$ and 50 mL of 1M $NaMnO_4$ were added to 882 mL of the caustic adjusted (1M) waste drained from the CUF. The precipitated waste was digested at 50°C for 4 hours. Table 3.1 lists the samples analyzed, mass dilution to be used for calculating DFs, and description of the sample.

Table 3.1. Samples and Mass Dilution for Calculating Decontamination Factors

Sample ID	Mass Dilution	Sample Description
MN-21	-	initial waste, filtered and acid digested
MN-22	1.1247	treated waste before CUF tests, filtered and acid digested
MN-23	1.1247	CUF filtrate during Condition 1, acid digested
MN-31	1.1247	CUF filtrate during Condition 6, acid digested
MN-32	1.1247	Composite CUF filtrate, Conditions 1-6, acid digested

The strontium, americium, and europium supernatant decontamination factors for samples MN-22 through MN-32 are shown in Table 3.2. All samples had very high decontamination for Sr and TRU components, greatly exceeding the requirements for ILAW. No decontamination of Co-60 or Cs-137 was observed. The concentration of Sr-90 and major TRU components for CUF composite filtrate, MN-32, is given to represent the expected Sr/TRU concentration of treated AN-107 waste.

Table 3.2. Strontium, Am, and Eu Decontamination Factors for Samples MN-22 to MN-32 and the Composition of Composite CUF Filtrate (MN-32)

	MN-22	**MN-23**	**MN-31**	**MN-32**	**Target DF**	**MN-32 (μCi/g)**
Sr-90	23	20	21	19	10	1.82
Am-241	32	20	19	17	5	8.9E-3
Eu-154	15	11	9	9	*	2.3E-2
Eu-155	15	11	8	9	*	1.7E-2

* reduces activity of ILAW

A separate sample of archived AN-107 was used for the lower reagent concentration, Sr/TRU removal test. The caustic level was adjusted by adding approximately 48 mL of 19M NaOH to 1-L of archived AN-107. The waste was well mixed and two samples removed. At ambient hot-cell temperature, approximately 57 mL of 1M$Sr(NO_3)_2$ and 34 mL of 1M $NaMnO_4$ were added to 1 L of the caustic adjusted (0.8M) waste. The precipitated waste was digested at 50°C for 4 hours. After cooling, the waste was filtered through a 0.45 um deadend filter and a sample of the filtrate collected. Table 3.3 lists the samples analyzed, mass dilution to be used for calculating DFs, and description of the sample.

Table 3.3. Samples and Mass Dilution for Calculating Decontamination Factors

Sample ID	**Mass Dilution**	**Sample Description**
MR-01	-	initial waste, acid digested (entrained solids present)
MR-02	-	initial waste, filtered and acid digested
MR-03	1.0929	treated waste filtrate, acid digested

Table 3.4 shows the DFs calculated based on the initial total sample (including entrained solids) and based on initial supernatant only for analyzed radioactive elements. Entrained solids removal contributed little to the DF. For Am-241, entrained solids account for approximately 10% of the total. The DFs were very high and consistent with the earlier Sr/TRU removal test at higher concentration. These results suggest that the decontamination requirements for Sr and TRU can be met with reduced reagent concentrations.

Table 3.4. Radioactive Element Decontamination Factors and Composition of Filtrate (MR-03)

	Total Sample DF	Supernatant DF	Target DF	Composition of Filtrate (uCi/g)
Sr-90	31	37	10	1.0
Am-241	18	10	5	1.3E-2
Eu-154	12	7	*	2.2E-2
Eu-155	12	7	*	1.5E-2
Total Beta	38	35	-	2.1

* reduces activity of ILAW

3.3 Change in Chemical Composition

The Sr/TRU precipitation and solids removal steps changed the chemical composition of the waste samples, i.e. solids and supernatant. Table 3.5 shows the compositional change of the major ICP elements and density for the various samples. The most interesting changes are for Fe and Mn. Both are relatively high in the initial waste (MN-21). On treatment, Fe removal was very high, and most likely correlated directly with the high removal of Am and Eu isotopes. Manganese also decreased significantly, which suggests that soluble Mn, likely Mn(II), is oxidized to insoluble Mn(IV) by reaction with Mn(VII). Calcium is precipitated on treatment while the Sr concentration increased because the original solution was below the saturation level for Sr, and a fraction of the added Sr remained soluble. The free hydroxide, determined by titration, decreased very little on precipitation with Sr and permanganate, 0.03M after correction for dilution. This is in contrast to the earlier proposed Fe precipitation for TRU removal, which consumed 3 times the added Fe concentration of hydroxide (3*0.075 = 0.225).

Sample MN-28 is the composition of the precipitated slurry, combined supernatant and precipitated solids. The solids were not analyzed, but comparison of the composition of the slurry to the initial composition and filtrates provides an indication of the solids composition. The solids contain primarily Sr and Mn as expected with lower amounts of Fe and Ca. Comparing the Fe concentration in the initial waste and slurry suggests entrained solids contain significant Fe.

Table 3.5. Concentration of Major ICP Metals in Samples MN-21 to MN-32 and Sample Density (data not corrected for sample dilution from added reagents)

	MN-21 (ug/g)	**MN-22 (ug/g)**	**MN-23 (ug/g)**	**MN-28 (ug/g)**	**MN-31 (ug/g)**	**MN-32 (ug/g)**
Ca	227	128	130	220	129	129
Fe	445	4.36	8	687	7.56	5.82
Mn	44.1	3.785	1.13	2810	0.48	0.5
Sr	1.14	112	115	4770	171	159
Na	104000	87900	84600	85200	84200	86200
	M	**M**	**M**	**M**	**M**	**M**
Na	5.68	4.69	4.49	4.60	4.47	4.58
OH^-	0.87	0.74	-	-	-	-
	g/mL	**g/mL**	**g/mL**	**g/mL**	**g/mL**	**g/mL**
density	1.26	1.23	1.22	1.24	1.22	1.22

The lower reagent concentration test included ICP data for initial waste with and without entrained solids, and for the filtrate. The concentration of the major ICP elements for these three samples in given in Table 3.6. The most significant difference between the data from the two different concentrations of added reagents is the higher soluble Mn in sample filtrate from the lower treatment level. The reason for this is not understood.

The composition of the entrained solids can be estimated by comparing the composition of MR-01, initial sample with entrained solids, and MR-02, initial sample filtered before analysis. The solids are rich in Fe and Mn. This is consistent with the data from Lumetta and Hoopes (1999) on washing/leaching of AN-107 solids.

Table 3.6. Concentration of Major ICP Metals in Samples MR-01 to MR-03 (data not corrected for sample dilution from added reagents)

	MR-01 (ug/g)	**MR-02 (ug/g)**	**MR-03 (ug/g)**
Ca	246	254	144
Cr	71.4	50.4	31.8
Fe	687	219	11.7
Mn	139	24.6	27.4
Sr	1.2	1.2	109
Na	92700	96000	104000
	(g/mL)	**(g/mL)**	**(g/mL)**
density	1.26	1.26	1.26

3.4 Sr/TRU Solids Removal

Removal of entrained solids from the archived AN-107 sample with crossflow filtration was not practical so the Sr/TRU precipitation was conducted with entrained solids present. The resulting slurry, containing both entrained solids and Sr/TRU precipitate, was transferred to the CUF and filtration tests conducted. Figure 3.1 shows filtrate flux data for entrained solids removal alone and for Sr/TRU precipitate/entrained solids removal from archived AN-107 at target conditions of 55 psi TMP and 12.2 ft/s crossflow velocity (Condition 1). The Sr/TRU removal process, precipitation with added $Sr(NO_3)_2$ and $NaMnO_4$, dramatically improved the filtrate flux rate for the archive AN-107 material. The filtrate flux was an order of magnitude higher for the treated waste, 0.11 gpm/ft^2 averaged over 30-60 min of testing.

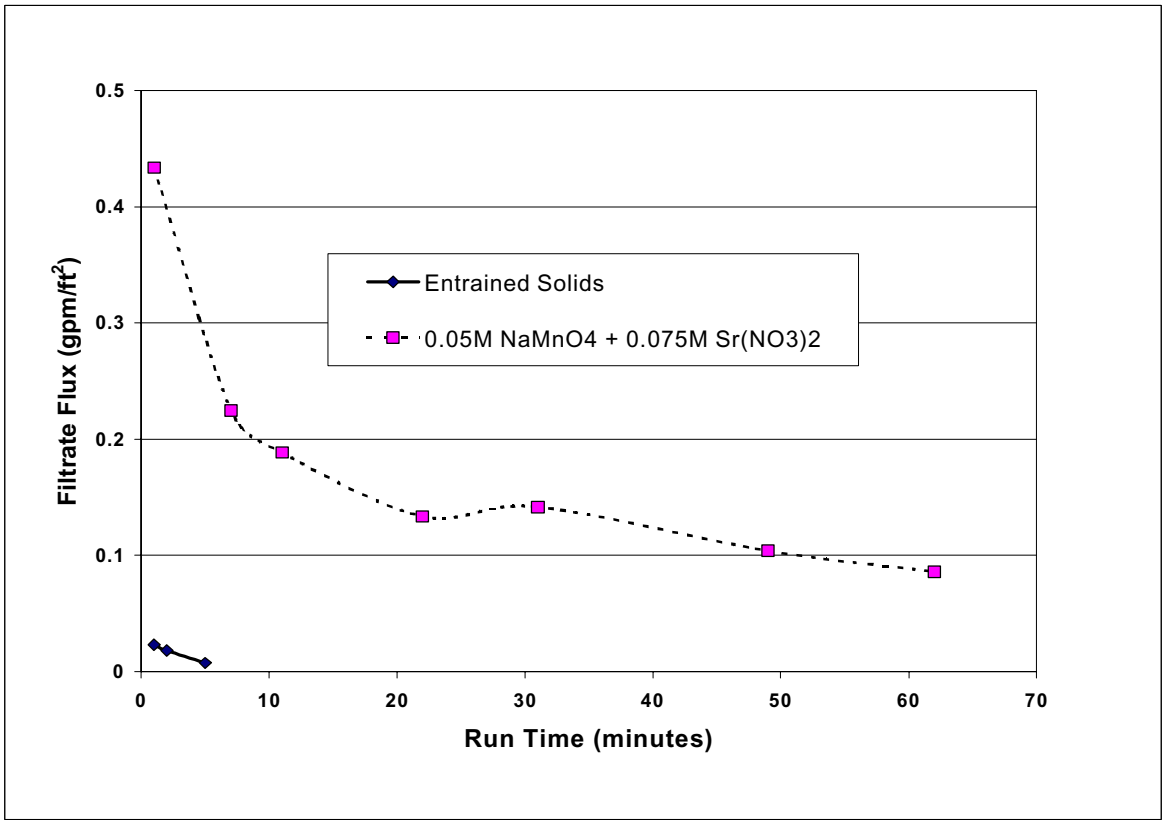

Figure 3.1. Comparison of the Filtrate Flux for Entrained Solids and Sr/TRU Precipitate/Entrained Solids Removal from Archived AN-107 Waste at Target Conditions of 55 psi TMP and 12.2 ft/s Crossflow Velocity

Based on these results, it appears that the Sr/TRU precipitate addition acts as a filter aid. This is further illustrated when the particle size distribution measurements are compared. The entrained solids and Sr/TRU precipitate samples were analyzed using the Microtrac UPA. The particles size distribution of the small particles measured with this instrument are shown in Figure 3.2. Results indicate that the bulk of the entrained solids are one micron and less, while the Sr/TRU precipitate particles are generally larger, beyond the range of the UPA instrument.

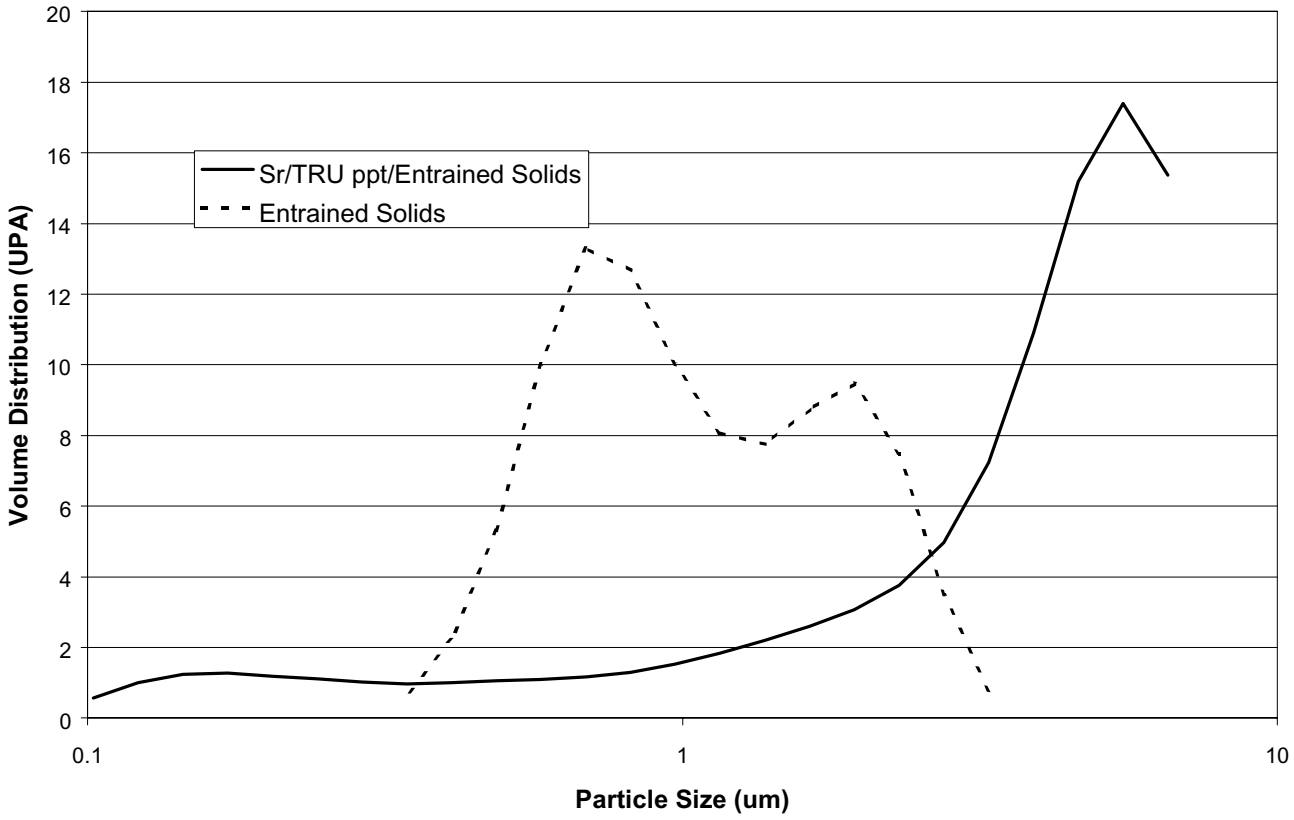

Figure 3.2. Volume-Weighted Particle Size Distribution Comparison Between Archive AN-107 Entrained Solids and Sr/TRU Precipitate

It is believed that during subsequent conditions (2-6), the shearing of the pump breaks apart the precipitated flocs resulting in smaller particles that reduce the filtrate flux. The results averaged between 20 and 60 minutes of testing for each condition are shown below in Figure 3.3. The results show a steady decline in filtrate flux over time. In fact, over conditions 2 through 5, it is nearly a linear decrease, in spite of the changes in transmembrane pressure and axial velocity. The pressure and velocity at the initial condition was repeated for the final condition. During the 8.5 hours of run time, the filtrate flux dropped by 55%.

The transmembrane pressure and axial velocity effects can also be seen in this data set. The transmembrane pressure appears to have little effect on the filtrate flux over the range from 38 to 70 psi. In fact, there is a decrease in filtrate flux between condition 2 (at 38 psid) and condition 3 (at 70 psid). The axial velocity, on the other hand, does appear to improve filtrate flux as evidenced by the increase in filtrate flux between condition 5 and condition 6. The reduction in filtrate flux for condition 3 compared to condition 2 may be largely due to the decreased axial velocity, not the increase in pressure.

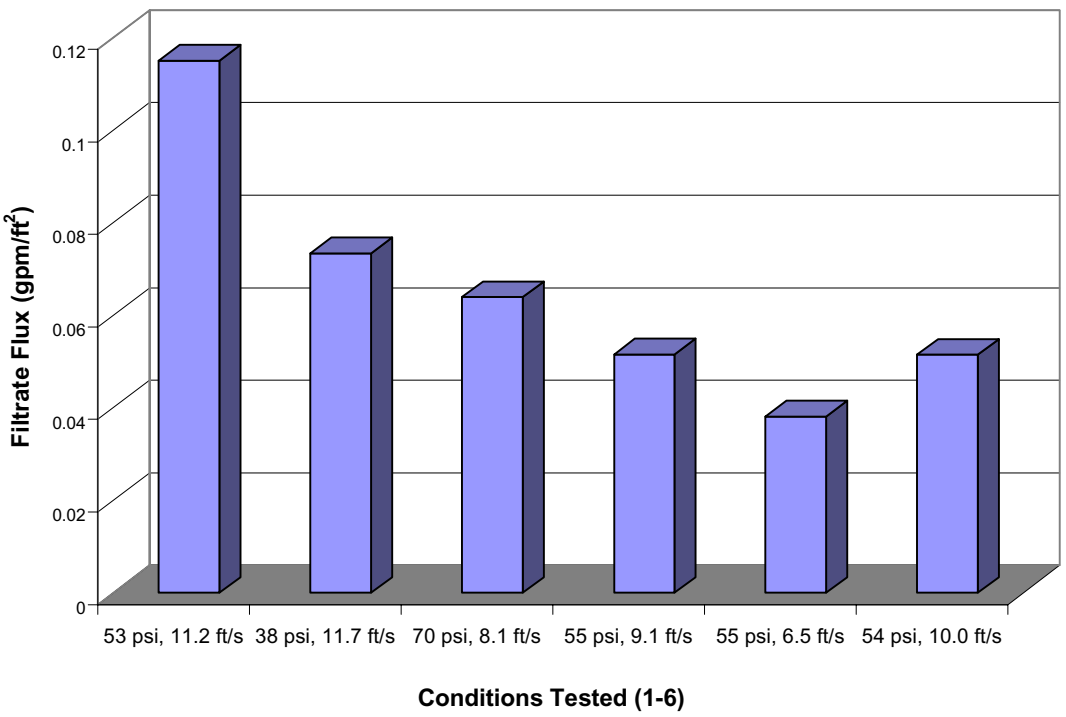

Figure 3.3. Average Filtrate Flux for Conditions Tested With the Archived AN-107 Sr/TRU Precipitate

In the full-scale process, the material will not continuously run through the pump to the extent seen in these liter-scale tests and thus filtrate fluxes may remain high at the other conditions. Thus, the time effect observed here may not significantly impact design. To remove the time dependency, statistical analysis is required. Non-linear regression was used to develop an equation describing flux as a function of time, axial velocity and transmembrane pressure. By minimizing the residual error, the following equation was developed:

$$Flux = 0.158e^{-time} + 0.00462 velocity + 0.000645 pressure - 0.0283$$

where flux is the filtrate flux in gpm/ft^2, time is hours from the start testing, velocity is the crossflow velocity in ft/s and pressure is the transmembrane pressure in psi. The time dependence is assumed to be exponential decay while the velocity and pressure dependence are assumed to be linear. Using this equation, it can be seen that both pressure and velocity increases result in improved filtrate flux. Crossflow velocity has a higher impact on filtrate flux over the conditions studied. Total filtration time is the dominant factor in predicting filtrate flux.

The cause of this reduction in filtrate flux over time may be the result of filter fouling. After cleaning the filter with DI water following the run, the filtrate flux was still significantly less than its original value. This is evidence of filter fouling. Furthermore, there is evidence that the particle size may have been reduced during the course of the run. These fines may become

lodged inside the filter and result in the reduction of filtrate flux. Particle size distribution was performed using a Microtrac UPA with the Sr/TRU precipitate taken after CUF filtration. These samples were analyzed with and without sonication. By sonicating the sample, the effects of shear, similar to that seen in the pump can be evaluated. A plot of the sample PSD before and after sonication is shown in Figure 3.4. The plot shows a reduction in particle size on sonication over the range evaluated. Particles below 0.1 um particle size were found.

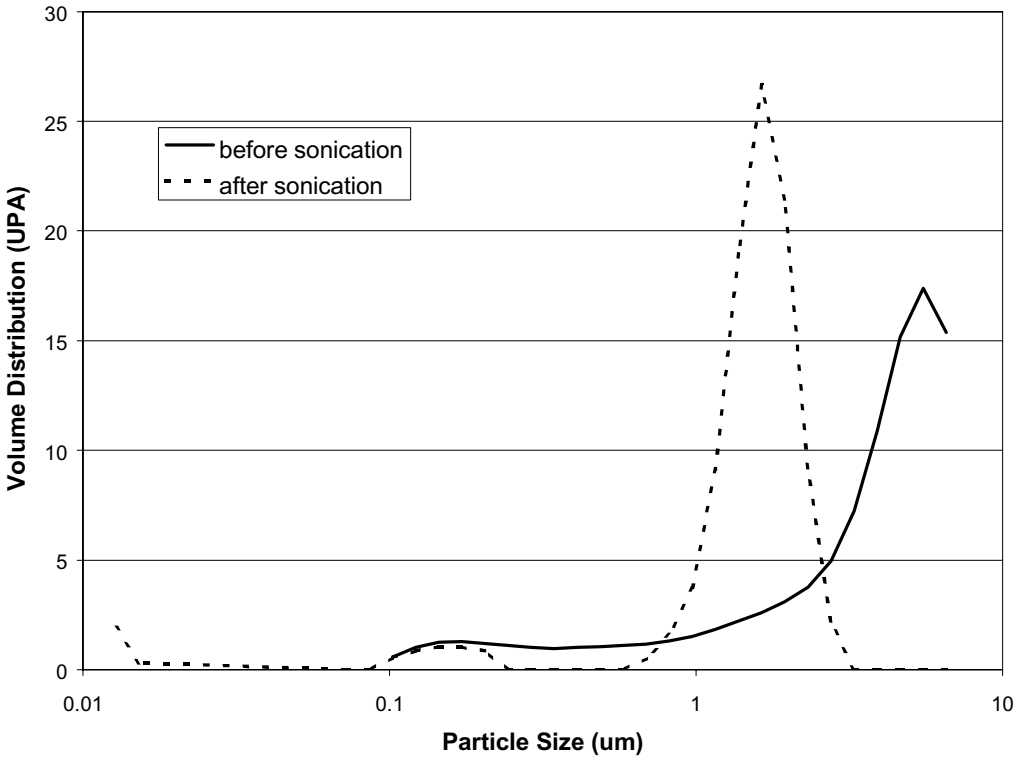

Figure 3.4. Particle Size Distribution Comparison of Archive AN-107 Sr/TRU Precipitate With and Without Sonication

4.0 CONCLUSIONS AND RECOMMENDATIONS

Archived AN-107 waste was used to evaluate entrained solids removal, Sr/TRU decontamination of supernatant, and Sr/TRU solids removal. Even though most of the entrained solids had been previously removed from the archived sample, the residual entrained solids rapidly fouled the filter element resulting in very poor filter performance. An attempt to run at higher pressure resulted in more fouling, and reduced filter performance. Filtration efforts to remove entrained solids were abandoned and the waste was treated for Sr/TRU removal with the entrained solids present.

The new processing scheme for Sr/TRU removal involving precipitation by added strontium and permanganate worked well. The decontamination factors for Sr and TRU components were significantly greater that the ILAW DF requirements for higher reagent concentrations of 1M hydroxide, 0.075M Sr, and 0.05M permanganate and lower reagent concentrations of 0.8M hydroxide, 0.05M Sr, and 0.03M permanganate. These results support the use of lower concentration of reagent additions in future tests. Optimization studies should be conducted to examine the reduction in added hydroxide from 1M to 0.5 M, reduction of Sr from 0.075M to 0.05M, and reduction in permanganate from 0.05M to 0.03M and the impact this reduction has on filtration performance with new samples from Tank AN-107.

The combined entrained solids and Sr/TRU precipitate were successfully filtered in the single element, crossflow filtration unit. The filtrate flux was high, >0.1 gpm/ft^2, at the initial test conditions of 53 psi and 11.2 ft/s for the treated archived AN-107 sample. The filter flux rate dropped significantly with time as testing progressed and appears to be a result of shearing the agglomerated solids and fouling of the filter element by the resulting fine particles. The relatively low clean water flux rates obtained at the end of the test also indicate filter fouling. Chemical cleaning was required to restore clean water flux rates to pre-test levels. The filter performance as a function of wt% solids could not be determined in this study because the treated waste volume was close to the minimum CUF volume and no dewatering could occur. Additional filtration tests need to be conducted to determine the filtrate flux as a function of wt% solids. Solids washing in the CUF also needs to be conducted to determine filtrate flux rate as a function of solids loading during washing.

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Appendix A: Test Instruction-041, Data Sheets, and Log Book Entries

Scoping Studies: Entrained Solids Removal, Permanganate Treatment for Sr/TRU Removal, and Precipitate Removal from Archived AN-107

Data Sheets

Log Book Entries

Appendix B: Test Instruction-063 and Log Book Entries

Sr/TRU Removal from Archived AN-107 with Minimal Reagent Addition

Log Book Entries

Appendix C: Analytical Data

Appendix D: Staff and Role/Responsibility

Staff Member	Role/Responsibility
Richard Hallen	Scientist/Technical Leader - Sr/TRU Precipitation
Kriston Brooks	Engineer/CUF System, Entrained Solids Removal, Sr/TRU Precipitation, and Precipitate Removal
Lynette Jagoda	Engineer Associate/CUF System, Entrained Solids Removal, Sr/TRU Precipitation, and Precipitate Removal
Gita Golcar	Scientist/Particle Size Analyses
Don Rinehart	Technician/Hot Cell Tests-Sr/TRU PPT/CUF Operation
Ralph Lettau	Technician/Hot Cell Tests-Sr/TRU PPT/CUF Operation
Dave Ortiz	Technician/Hot Cell CUF Operation and Cleaning
Vaughn Hoopes	Technician/Hot Cell sample prep.
Mac Zumhoff	Technician/Hot Cell Operations

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Appendix A: Test Instruction-041, Data Sheets, and Log Book Entries

Scoping Studies: Entrained Solids Removal, Permanganate Treatment for Sr/TRU Removal, and Precipitate Removal from Archived AN-107

Data Sheets

Log Book Entries

RT HALLEN
Date 6/10/99
Route
File TI-041
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Task 2.3 & 2.5
29953

PNNL Test Instruction

Document No.: BNFL-TP-29953-041
Rev. No.: 0

Title: Scoping Studies: Entrained Solids Removal, Permanganate Treatment for Sr/TRU Removal, and Precipitate Removal from Archived AN-107 (previously treated by Cs IX)

Work Location: RPL SFO HLRF	**Page** 1 of 15
Author: Richard T. Hallen	**Effective Date:** Upon Final Approval **Supercedes Date:** New
Use Category Identification: Information	
Identified Hazards: ___Radiological ___ Hazardous Materials ___ Physical Hazards ___ Hazardous Environment ___ Other:	**Required Reviewers:** _X_ Technical Reviewer ____ SFO Manager

Are One-Time Modifications Allowed to this Test Instruction? _X_ Yes ___ No

NOTE: If Yes, then modifications are not anticipated to impact safety. For documentation requirements of a modification see SBMS or the controlling Project QA Plan as appropriate.

On-The Job Training Required? ____ Yes or _X_ No

FOR REVISIONS:
Is retraining to this procedure required? ____Yes _X_ No

Does the OJT package associated with this procedure require revision to reflect procedure changes?
____Yes ____No ___X___N/A

Approval	Signature	Date
Author	Richard T. Hallen	6/9/99
Technical Reviewer	H T Brooks	6/9/99

Uncontrolled Copy
- see 0203 Brooks

1.0 Applicability

This test instruction is to be used to perform studies on entrained solids removal, permanganate treatment for Sr/TRU removal and precipitate solids removal from archived AN-107 waste that has been previously treated by cesium ion exchange resin. The entrained solids and precipitate will be removed using the Cell Unit Filter (CUF) in the HLRF A-cell from approximately 1-L of AN-107 waste (C3E5, C3E4 and/or C3E6). The permanganate treatment is to be conducted after the entrained solids removal and before precipitate removal.

2.0 Supporting Documents

This test instruction is not a stand-alone document. It will be used in conjunction with PNNL Operating Procedure BNFL-TP-29953-020 which contains the necessary procedural information for the safe operation of the CUF. It is also linked to PNNL Test Plan #s BNFL-TP-29953-004 and -013 which contains an overall description of the project, ES&H compliance, emergency response, and the hazards assessment and mitigation. Scoping tests on the permanganate treatment of AN-107(cesium removed) is described in detail in test instruction, TP-29953-037.

3.0 Responsible Staff

The staff responsible for executing this test plan are as follows.

- Task Managers – Kriston Brooks and Rich Hallen
- SFO Manager – Randy Thornhill
- Test Scientists/Engineers – Kriston Brooks, Ken Rappe, Lynette Jagoda, Sam Bryan, and Rich Hallen
- Hot Cell Technician –Mac Zumhoff, Don Rinehart, and Ralph Lettau
- Radiological Control Technician

4.0 Materials, Equipment, Supplies and Reagents Needed

4.1 Materials Required

1. Twenty two 20 mL glass scintillation vials for filtrate and slurry samples, pre-labeled on top and side as follows: MN-21 through MN-30 and CUF-AAN107-01 through CUF-AAN107-10, and Tc IX –01 and Tc IX-02.
2. Three 1 liter polyethylene bottles. They should be labeled as follows: "MNO4 CUF Slurry," "CUF Archive AN-107 First Rinse" and "MNO4 CUF First Rinse." The bottle labeled "MNO4 CUF Slurry" should be marked with graduation lines at 100 mL intervals up to 1000 mL.
3. Two 10 liter containers, one labeled for the alkaline rinses and the other labeled for the acidic rinses.
4. Containers for draining from the bottom of the pump and from the sample valve.
5. 12 liters of 0.2 micron filtered DI water for determining clean water flux and for rinsing the CUF

4.2 Equipment

1. 4000 gram balance
2. pH paper

3. Hand held camera. To be used to read filtrate flowmeter.
4. Stopwatch
5. Calculator
6. CUF Ultrafiltration system with 100 mL plug in place using 0.1 micron Mott-L filter and new pump rotor
7. 1000 W Chiller
8. 2-L flask
9. hot-stir plate (B-cell)
10. big stir bar
11. 2 mL pipet

4.3 Reagents Needed

1. 2 liters of 1M HNO_3 + 0.1-0.2M Citric Acid
2. 1 liter of 5 ppm hypochlorite solution (pH > 7)
3. 50 mL of 1.0M NaMnO4
4. 75 mL of 1.0M Sr(NO3)2
5. 1 liter of Archive AN-107 that has been adjusted to 1M OH

4.4 Other Supplies

1. Workplace Copy of Operating Procedure BNFL-TP-29953-020
2. Extra Copies of Data Sheets 1, 2, and 3
3. Laboratory Record Book
4. DAS disk for recording data

" BNW-13745 "

5.0 Test Instructions for CUF Operation and Permanganate Treatment

The laboratory record book (LRB) shall be used to record other testing information as required by this procedure and all test conditions not stated by this procedure.

Cross-contamination between samples and contamination of samples from outside sources must be minimized at each step. Use new tools and bottles for each sample as much as possible. Those tools that are reused should be washed and rinsed prior to reuse.

Keep all test materials in sealed containers as much as possible to prevent them from drying.

5.1 Pre-start for Scoping Studies

5.1.1 Inventory materials, equipment, supplies, and reagents to ensure all required items are available. Assure that all materials have been modified for remote handling.

5.1.2 Do the following and initial and date when each item is completed.

_____ Review PNNL Operating Procedure BNFL-TP-29953-020.

_____Review the work instructions in BNFL-TP-29953-041.

5.1.3 Conduct the "0.0 Pre-Start" operations in BNFL-TP-29953-020. Drain the system overflow container.

5.1.4 Perform "1.0 Start-Up" operations in BNFL-TP-29953-020 with 1 liter of filtered DI water with one variation: V4 the filtrate control valve should be closed. Run CUF for 5 minutes at between 4-6 gpm. In-line pressure should be varied from using V1 from 10 to 70 psig. Ensure that there are no leaks in the system. If leaks are detected, shut down system immediately.

5.1.5 Conduct the "10.0 Draining the system" operation in BNFL-TP-29953-020.

5.1.6 Perform "1.0 Start-Up" operations in BNFL-TP-29953-020 with 1.0 liter of filtered, distilled water with one variation: V4 the filtrate control valve should be closed. Run CUF for 5 minutes at between 4-6 gpm. In-line pressure should be varied from using V1 from 10 to 70 psig.

5.1.7 Conduct the "10.0 Draining the system" operation in BNFL-TP-29953-020.

5.1.8 Perform "1.0 Start-Up" operations in BNFL-TP-29953-020 with 1.0 liter of filtered, distilled water

5.1.9 Perform "6.0 Back pulsing" operations in BNFL-TP-29953-020.

5.1.10 Shut off the system and conduct the "10.0 Draining the system" operation in BNFL-TP-29953-020.

5.1.11 Perform "1.0 Start-Up" operations in BNFL-TP-29953-020 with 1.0 liter of filtered, distilled water.

5.1.12 Conduct the "3.0 Operation during Ultrafilter Recycle Mode" operations in BNFL-TP-29953-020 using the conditions below. Filtrate flow rate should be monitored and data collected in the operating procedure. Each test should be performed for only 20 minutes and the system should be back pulsed. After each condition, the test engineer should initial and date the table below.

Condition	Flowrate (gpm)	Transmembrane Pressure (psig)	Initial and date when complete
1	4.20	10	7/16/99
2	4.20	20	7/16/99
3	4.20	30	7/16/99

5.1.13 Shut off the system and conduct the "10.0 Draining the system" operation in BNFL-TP-29953-020.

5.2 Start-Up for CUF Operation

5.2.1 Obtain the following information:

M&TE List:

PM4000 Balance 1:

Calib ID 362-06-01-054 Calib Exp Date 8/99

Location A-cell North

PM420 Balance 2:

Calib ID 362-06-01-061 Calib Exp Date 8/99

Location A-Cell South

Thermocouple:

Calib ID _____ Calib Exp Date _____

Location _____ Thermocouple type _____

Digital Thermometer:

Calib ID _____ Calib Exp Date _____

Location _____

5.2.2 Record the tare and sample weight of AN-107 with storage bottles (verify/identify sample is AN-107 id#). (This bottle should have had 40 grams of NaOH pellets in it to raise the hydroxide concentration to 1M.) Record the weight(s) below.

Bottle Label Archive AN-107 w/ 1M NaOH

Tare bottle 127.82 g NaOH wt 39.98g

Bottle and sample ~~1415.77~~ g akg 6/9/99

1410.42 akg 7/15/99

1242.62 g of Archived AN-107 - check density of MN-01 1.279 = 978.6

5.2.3 Conduct the "1.0 Start-Up" operations in BNFL-TP-29953-020 using AN-107. Shake the waste thoroughly before adding it to the slurry reservior. There may be some solids left the jars that cannot be transferred by shaking. If so, consult with the cognizant engineer on recovering these solids. DI water addition or scraping the sides with a spatula could be attempted. Record the method of recovery in the LRB.

5.2.4 Record the weight(s) of empty reaction vessel or sample bottles in the spaces provided below.

Empty bottle _____ g

ADDED _____ g
TOTAL _____ g

5.2.5 Record the level in the slurry reservoir sight glass.

Height ~~6~~ _____ inches

5.3 CUF Operation: Entrained Solids Removal Test with AN-107

5.3.1 Conduct the "3.0 Operation during Ultrafilter Recycle Mode" operations in BNFL-TP-29953-020 using the conditions below. Filtrate flow rate should be monitored and data collected as specified in the operating procedure. After each condition, the test engineer should initial and date the table below. If no filtrate flow occurs or the filtrate flow is less than 10 mL/min, discontinue testing and move to the next condition. After each condition, the test engineer should initial and date the table below.

NOTE: Samples of filtrate should be taken within the first 10 minutes of operation during Condition 1 and 30 minutes into each of the other 5 conditions. A slurry sample should be taken during Condition 3. Instructions for sampling are provided below.

NOTE: Test conditions below are suggested and all conditions do not need to be run, make sure condition 1 is run for 1 hour, other conditions should be run at least 20 minutes, run as many of the conditions as time allows.

Condition	Flowrate (gpm)	Transmembrane Pressure (psig)	Initial and date when complete
1	4.20	55	
2	4.20	40	
3	4.20	70	
4	3.13	55	
5	5.23	55	
6	4.20	55	

Not run- filter plugs

5.3.2 Obtain 1 filtrate sample of at least 10 grams within the first 10 minutes of operation (during Condition 1). Record the weight and sample number in Data Sheet 3. These will be used for chemical and radiochemical analyses.

5.3.3 Obtain filtrate samples of at least 5 grams each following "8.0 Filtrate Sampling" in BNFL-TP-29953-020 and using the pre-labeled sample vials after approximately 30 minutes of operation for each condition. Record the weight and sample number in Data Sheet 3. These will be used for chemical and radiochemical analyses.

Tare wts
CUF-AN107-01 17.0259
CUF-AN-107-02 17.0349

with Sample
25.580

5.3.4 Obtain 2 slurry samples of at least 10 grams following "7.0 Slurry Sampling" in BNFL-TP-29953-020. The samples should be taken after condition 3. The first slurry sample should not be saved, but dumped back into the tank. Record the weight and sample number in Data Sheet 3.

5.3.5 Tare weigh a prelabeled 20 mL sample vial.

5.3.6 Using a pipet, transfer 2 mL from each of the 6 filtrate sample vials taken previously in step 5.3.3 into the sample vial. Record the weight and sample number in Data Sheet 3. This composite will be used for chemical and radiochemical analyses.

5.3.7 Conduct the "11.0 Shutting down" operation in BNFL-TP-29953-020.

5.4 Entrained Solids Removal Test: Rinsing and Draining the System

5.4.1 Tare the 1 liter bottle labeled, ~~"MNO4 CUF Slurry."~~ Back into original Bottle

Weight of bottle and lid ~~Slurry~~ 1290.86 g

but volume almost same as put into system wt. loss prob. mostly solids removed.

5.4.2 Conduct the "10.0 Draining the system" operation in BNFL-TP-29953-020. Collect liquid in 1 liter bottle. Make sure as much materials as possible has been collected. This materials will be used for the permanganate treatment. Weigh bottle after all liquid has been removed.

Weight of slurry, bottle and lid _____ g

Weight of material collected _____ g

NOTE: Sections 5.5, 5.6 and 5.7 can be completed after Step 5.4.2

5.4.3 Conduct the "9.0 Rinsing the system" operation in BNFL-TP-29953-020. The first rinse should be done with 1 liter of ~~distilled water~~ 1.2 M NaOH. This liquid should be collected and saved in the container labeled "CUF Archive AN-107 First Rinse." The second rinse should be done with 2 liters of filtered, distilled water, and the final rinse with 1 liter filtered, distilled water. The second and third rinses should be collected separately from the first in the alkaline rinse storage container. Conduct the acid wash of the CUF unit with 1M HNO3/0.1-0.2M Citric Acid as described in the CUF operating procedure. The acidic solutions should be allowed to sit in the CUF overnight. When drained, the acidic solutions should be placed in a separate container.

5.4.4 The CUF should be drained according to "10.0 Draining the system" operation in BNFL-TP-29953-020 and rinsed at least 3 times with filtered, distilled water to bring the pH back up to neutral. The acidic solutions should be placed in a separate container from the alkaline ones.

5.4.4 Perform "1.0 Start-Up" operations in BNFL-TP-29953-020 with 1.0 liter of filtered, distilled water.

5.4.5 Perform "6.0 Back pulsing" operations in BNFL-TP-29953-020.

5.4.6 Shut off the system and conduct the "10.0 Draining the system" operation in BNFL-TP-29953-020.

5.4.7 Perform "1.0 Start-Up" operations in BNFL-TP-29953-020 with 1.0 liter of filtered, distilled water.

5.4.8 Conduct the "3.0 Operation during Ultrafilter Recycle Mode" operations in BNFL-TP-29953-020 using the conditions below. Filtrate flow rate should be monitored and data collected in the operating procedure. Each test should be performed for only 20 minutes and the system should be back pulsed. After each condition, the test engineer should initial and date the table below.

Condition	Flowrate (gpm)	Transmembrane Pressure (psig)	Initial and date when complete
1	4.20	10	
2	4.20	20	
3	4.20	30	

5.4.9 Shut off the system and conduct the "10.0 Draining the system" operation in BNFL-TP-29953-020.

5.5 Sr/TRU Removal by Permanganate Treatment

NOTE: Sections 5.5, 5.6 and 5.7 can be completed after Step 5.4.2

5.5.1 Setup the ppt reaction equipment as specified. The precipitation is to be conducted in a 2L flask. Secondary containment should be used to allow recovery from a possible breach of the 2 L flask. Place stir bar in flask.

Shake the bottle of waste thoroughly and collect two 20 mL one in the vial labeled Tc IX-01 and one in MN-21. Shake and transfer the remaining waste to the reaction vessel. There should be some solids. If solids are observed, record observation or notify the cognizant scientist/engineer (record observations in the LRB).

5.5.2 Record the weight(s) of the empty bottle with lid in the spaces provided below. Calculate the amount of material transferred to the reaction vessel. The cognizant scientist/engineer will calculate the amount of strontium and permanganate solution to add.

AN-107

Full 1239.16 g Full ____ g
Empty 1345 g Empty ____ g

900 mL mark on 2 liter flask

Added 1107.71 g Added ____/____ g Total ____/____ g

Density = ____ g/mL total volume added ____ mL

5.5.3 Calculate the amount of strontium and permanganate solution to add . The cognizant scientist/engineer calculations are to be reviewed prior to the additions, and retained in the project files.

75 mL $Sr(NO_3)_2$ (1M)

Record here and below 50. mL Density ____ g/mL Weight ____ g

↳ $NaMnO_4$ (1M)

5.6 Permanganate Treatment of AN-107

RJH 7/26/99

5.6.1 Turn on stirrer. ~~Turn heat on and heat to 50 +/- 5 C.~~ Do not heat!

5.6.2 While continuously stirring the waste sample, personnel are to slowly add 75 mL of 1.0 M $Sr(NO_3)_2$ solution per each liter of AN-107 sample. (based upon BNF-003-98-0023, add 1M Sr at a rate of 2 to 20 mL/min/L of waste, target 10 mL/min/L of waste) - add over 5-10 minute period - then stir for additional 5-10 minutes until well mixed. RJH 7/26/9

Tare bottle of 1M Sr 184.3352 g
Tare Empty bottle 100.522
Weight Added 83.813

~~5.6.4 Heat the waste mixture at 50 ± 5°C with stirring for 2 hours after completing the addition of the Sr.~~ RJH 7/26/99

5.6.5 Slowly add 50 mL of 1 M $NaMnO_4$ solution per each liter of AN-107 sample. (based upon BNF-003-98-0023, add 1 M $NaMnO_4$ at a rate of 2 to 20 mL/min/L of waste, target 10mL/min/L of waste) add over a 5-10 minute period - then stir for an additional 5-10 minutes until well mixed. RJH 7/26/99

Tare bottle of 1M NaMnO4 112.1823 g
Tare Empty bottle 57.857 g
Weight Added 54.325 g

4 RJH 7/26/99

5.6.6 Heat the waste mixture at 50 ± 5°C with stirring for 2 hours after completing the addition of the $NaMnO_4$.

5.6.6 Turn off the stirrer and allow the waste to cool to 25 ± 5°C. If possible, use a video recorder to document the settling behavior of the waste mixture. Record the volume of settled solids. Not performed RJH 8/3/99

5.6.7 ~~Stir waste and~~ collect two 20 mL samples, one in Tc IX-02 and one in MN-22. - of supernat after cooling/settling.

5.7 Completion of Permanganate Treatment/Precipitation and Startup of CUF Testing

(The solids removal from the reaction vessel is to occur as soon after precipitation as possible. If for some reason, a delay is required between precipitation and solids removal, transfer the slurry from the reaction vessel to 1 or 2 - 1 liter poly bottles as needed.)

5.7.1 Conduct the "1.0 Start-Up" operations in BNFL-TP-29953-020 using permanganate treated AN-107. Record the weight of the reaction vessel and ppt. Shake/mix the waste slurry thoroughly before transferring to the slurry reservior. There may be some solids left in the flask that is difficult to transfer. If so, consult with the cognizant scientist/engineer on recovering these solids. DI water addition/washing could be attempted if the amount of solids looks significant. Record the method of recovery in the LRB.

5.7.3 Record the weight(s) of empty reaction vessel or sample bottles.

Vessel and ppted AN-107 2204.704 g
Empty vessel 1024.34 g
ADDED 1180.40 g

5.7.3 Record the level in the slurry reservoir sight glass.

Height Not seen inches

5.8 CUF Operation: Sr/TRU Precipitate Removal Test with Archived AN-107

5.8.1 Conduct the "3.0 Operation during Ultrafilter Recycle Mode" operations in BNFL-TP-29953-020 using the conditions below. Filtrate flow rate should be monitored and data collected as specified in the operating procedure. After each condition, the test engineer should initial and date the table below. If no filtrate flow occurs or the filtrate flow is less than 10 mL/min, discontinue testing and move to the next condition. After each condition, the test engineer should initial and date the table below.

NOTE: Samples of filtrate should be taken within the first 10 minutes of operation during Condition 1 and 30 minutes into each of the other 5 conditions. A slurry sample should be taken during Condition 3. Instructions for sampling are provided below.

NOTE: Test conditions below are suggested and all conditions do not need to be run, make sure condition 1 is run for 1 hour, other conditions should be run at least 20 minutes, run as many of the conditions as time allows.

Condition	Flowrate (gpm)	Transmembrane Pressure (psig)	Initial and date when complete
1	4.20	55	7/27/99
2	4.20	40	7/27/99
3	4.20	70	7/27/99
4	3.13	55	7/27/99
5	5.23	55	

6	4.20	55	

5.8.2 Obtain 1 filtrate sample of at least 10 grams within the first 10 minutes of operation (during Condition 1). Record the weight and sample number in Data Sheet 3. These will be used for chemical and radiochemical analyses.

5.8.3 Obtain filtrate samples of at least 5 grams each following "8.0 Filtrate Sampling" in BNFL-TP-29953-020 and using the pre-labeled sample vials after approximately 30 minutes of operation for each condition. Record the weight and sample number in Data Sheet 3. These will be used for chemical and radiochemical analyses.

5.8.4 Obtain 2 slurry samples of at least 10 grams following "7.0 Slurry Sampling" in BNFL-TP-29953-020. The samples should be taken after condition 3. The first slurry sample should not be saved, but dumped back into the tank. Record the weight and sample number in Data Sheet 3.

5.8.5 Tare weigh a prelabeled 20 mL sample vial.

5.8.6 Using a pipet, transfer 2 mL from each of the 6 filtrate sample vials taken previously in step 5.8.3 into the sample vial. Record the weight and sample number in Data Sheet 3. This composite will be used for chemical and radiochemical analyses.

5.8.7 Conduct the "11.0 Shutting down" operation in BNFL-TP-29953-020.

5.9 Sr/TRU Precipitate Removal Test: Rinsing and Draining the System

5.9.1 Tare the 1 liter bottle labeled, "MNO4 CUF Slurry."

Weight of bottle and lid _____ g

5.9.2 Conduct the "10.0 Draining the system" operation in BNFL-TP-29953-020. Collect liquid in 1 liter bottle. Make sure as much materials as possible has been collected. This materials will be used for the permanganate treatment. Weigh bottle after all liquid has been removed.

Weight of slurry, bottle and lid _____ g

Weight of material collected _____ g

~850 ml received

5.9.3 Conduct the "9.0 Rinsing the system" operation in BNFL-TP-29953-020. The first rinse should be done with 1 liter of distilled water. This liquid should be collected and saved in the container labeled "MNO4 CUF First Rinse." The second rinse should be done with 2 liters of filtered, distilled water, and the final rinse with 1 liter filtered, distilled water. The second and third rinses should be collected separately from the first in the alkaline rinse storage container. Conduct the acid wash of the CUF unit with 1M HNO3/0.1-0.2M Citric Acid as described in the

CUF operating procedure. The acidic solutions should be allowed to sit in the CUF overnight. When drained, the acidic solutions should be placed in a separate container.

5.9.4 The CUF should be drained according to "10.0 Draining the system" operation in BNFL-TP-29953-020 and rinsed at least 3 times with filtered, distilled water to bring the pH back up to neutral. The acidic solutions should be placed in a separate container from the alkaline ones.

5.9.4 Perform "1.0 Start-Up" operations in BNFL-TP-29953-020 with 1.0 liter of filtered, distilled water.

5.9.5 Perform "6.0 Back pulsing" operations in BNFL-TP-29953-020.

5.9.6 Shut off the system and conduct the "10.0 Draining the system" operation in BNFL-TP-29953-020.

5.9.7 Perform "1.0 Start-Up" operations in BNFL-TP-29953-020 with 1.0 liter of filtered, distilled water.

5.9.8 Conduct the "3.0 Operation during Ultrafilter Recycle Mode" operations in BNFL-TP-29953-020 using the conditions below. Filtrate flow rate should be monitored and data collected in the operating procedure. Each test should be performed for only 20 minutes and the system should be back pulsed. After each condition, the test engineer should initial and date the table below.

Condition	Flowrate (gpm)	Transmembrane Pressure (psig)	Initial and date when complete
1	4.20	10	
2	4.20	20	
3	4.20	30	

5.9.9 Shut off the system and conduct the "10.0 Draining the system" operation in BNFL-TP-29953-020.

5.10 Experimental Clean Up and Sample Disposition.

Clean up all of the equipment used.

Do not discard any samples without written instructions from the task manager. Duplicate samples were collected at all sampling points, but only primary samples submitted for analyses. The duplicates are to be retained until review of the analytical data is completed and notification is writing is received to dispose of these samples. The alkaline and acid CUF wash solutions should be disposed of properly.

6.0 Sample Analysis

The point of contact for physical property sample analysis of the slurry samples is Paul Bredt. The point of contact for the sample analysis of the filtrate, wash, and filtered solids samples is Mike Urie and Rick Steele.

6.1 Chemical and Radiochemical Analysis

The points of contact for the sample analysis of the slurry and filtrate samples are Mike Urie and Rick Steele.

The following samples should be transferred to the SAL hot cells for prep work and analysis for the entrained solids test if the average filtrate flow at any condition is >40 mL/min:

- One sample of slurry from CUF described in Step 5.3.4
- Initial sample of filtrate taken during Condition 1 described in Step 5.3.2
- Final sample of filtrate taken during Condition 6 described in Step 5.3.3
- Composite filtrate prepared in Step 5.3.6

The following will be performed on the above samples if they are analyzed: TOC/TIC on the original sample, then acid digest followed by GEA, Sr-90 analysis and ICP-AES.

The two samples labeled Tc IX-01 and Tc IX-02 should be transfer to the SAL and Dean Kurath notified of their arrival. These samples will be used for batch contacts with Tc ion exchange resins.

The two 20g samples, MN-21 and MN-22 should be transferred to the SAL hot cells for prep work and analysis for the Sr/TRU precipitation test. Once in SAL the samples should be filtered with 0.45 micron disposable syringe filter as soon as possible (or filter in A-cell when taken).

The following samples should be transferred to the SAL hot cells for prep work and analysis from the Sr/TRU Precipitate Removal test:

- Initial sample of filtrate taken during Condition 1 described in Step 5.8.2 (ICP, Sr and GEA)
- Final sample of filtrate taken during Condition 6 described in Step 5.8.3 (ICP, Sr and GEA)
- Composite filtrate prepared in Step 5.8.6 (ICP, Sr and GEA)
- One sample of slurry from CUF described in Step 5.8.4 (acid digest-ICP, Sr and GEA)

Table 3 below shows the sample analysis list for the Sr/TRU precipitation and SR/TRU solids removal test. The table lists the analyses to be performed on samples generated from this test instruction.

Table 3. Samples and Their Required Analyses (if vial numbers differ, cross out and show actual 3)

Process Variable	Vial ID	Sample Type	Sample Preparation	Analysis Description[a]
AN-107 after entrained solids	MN-21	as received	0.45 um filter, acid digest	Sr/Am, [OH⁻]
AN-107 after Sr/TRU PPT	MN-22	ppted waste	0.45 um filter, acid digest	TOC/TIC, Sr/Am, ICP, [OH⁻]
1st CUF Permeate Sr/TRU PPT	MN-23	CUF filtrate	acid digest	Sr/Am, ICP
Final CUF Permeate	MN-24	CUF filtrate	acid digest	Sr/Am, ICP
Composite CUF Permeate	MN-25	Filtrate	acid digest	Sr/Am, ICP
Final CUF Slurry	MN-26	Slurry	acid digest	Sr/Am, ICP

(a) Descriptions of analyses are contained in Table 4.

Table 4. Description of Analyses

Constituent	Analysis Method	PNNL Procedure No.
Americium-241, Eu isotopes	GEA	PNL-ALO-450
Strontium-90 (Yttrium-90)	Separations and Beta Counting	PNL-ALO-476/431
Hydroxide	EPA SW-846 Modified Method, 310(3)	PNL-ALO-228
TOC/TIC		
Metal Ions (see Table 5 list)	ICP-AES	PNL-ALO-211/280

Table 5. Analytical Requirements for Supernate/Filtrate and Centrifuged Solids

Analyte	Centrifuged Solids Minimum Reportable Quantity microCi/gm	Supernate/Filtrate Minimum Reportable Quantity microCi/ml	Analysis Method
Strontium-90	7.01E+01	1.5E-01	Chemical Separation & Beta Count
Americium-241	1.2E-03	7.2E-04	GEA
	microgm/gm	microgm/ml	
Al	3.3E+02	7.5E+01	Acid Digestion followed by ICP-AES
Ba	6.0E+02	7.8E+01	
Ca	1.8E+02	1.5E+02	
Cd	1.1E+01	7.5E+00	
Co	3.0E+00	3.0E+01	
Cr	1.2E+02	1.5E+01	
Cu	1.8E+01	1.7E+01	
Eu	NA	NA	
Fe	1.4E+02	1.5E+02	
K	1.5E+03	2.0E+02	
La	6.0E+01	3.5E+01	
Mg	5.4E+02	1.5E+02	
Mn	3.0E+02	1.5E+02	
Mo	6.0E+00	9.0E+01	
Na	1.5E+02	7.5E+01	
Ni	1.6E+02	3.0E+01	
Pb	6.0E+02	3.0E+02	
Si	3.0E+03	1.7E+02	
Sr	3.0E+02	8.7E+01	
Ti	1.5E+02	1.7E+01	
U	6.0E+02	6.0E+02	
Zn	6.0E+00	1.65E+01	
OH-		0.05M	

Data Sheet 1: Operating Data

TJ 8/3/99

Task 2.3 & 2.5

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p. 1 of 5

See TP-29953-041

Date: July 16, 1999
Tank Number: Water Test
Filter: Second Matt-6 0.1µm
Test Conditions: F = 4.2 gpm test 1 = 10 psig test 2 = 20 psig test 3 = 30 psig
Operator: D. Rinhart
Test Engineer: LK Jagoda & KP Brooks

Test No.	Time	Chiller Temp.	Slurry Temp.	Slurry loop Flowrate	Filter Outlet Pressure	Permeate Pressure	Filter Inlet Pressure	Filtrate Flowrate Volume	Filtrate Flowrate Time	Filtrate Flowrate Flowrate	Tank Level	Comments
2	13:03		23.5	3 gpm	Not operating	2	20					Rotameter = 30
2	13:08						20					Rotameter = 26
2	13:16						18					Rotameter = 24
2	13:23					e	18					Rotameter = 25
2	13:32			4 gpm		2	20					Rotameter = 27
2	13:49	25.5	26.7			3	20					Rotameter = 27
2	13:57	25.5	24.6	4 gpm		3	20					R = 25
2	14:02	23.8	22.5	4 gpm		3	20					R = 24
2	14:03	23.5	22.1	4 gpm			20					R = 24
1	14:12	23.5	22.7	~4 gpm			10					R = 10
1	14:15	24.0	23.4	~4 gpm			10					R = 10
1	14:19	24.7	24.2	~4 gpm			~9					R = 9
1	14:22	25.4	24.9	~4 gpm			10					R = 10
1	14:26	25.9	25.4	~4 gpm			9.5					R = 10
1	14:30	25.5	24.6	~4 gpm			9.5					R = 9
1	14:32	25.0	23.7	~4 gpm			9.5					R = 9
3	14:40	23.2	22.2	~4 gpm		4	30.0					R = 45
3	14:42	23.9	23.2	~4 gpm		4	30.0					R = 40
3	14:47	25.0	24.5	~4 gpm		3	30.0					R = 39 adj
3	14:53	26.1	25.5	~4 gpm		3	30.0					R = ~~38 39~~ 38
3	15:00	24.3	23.0	~4 gpm		3	30.0					R = 37

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Task 2.3 & 2.5
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Data Sheet 1: Operating Data

Date: July ~~26~~ 23~~19~~, 1999
Tank Number: Water test After AN-107 Clean up
Filter:
Test Conditions: Test 2 = 20 psig ~4.2 slpm Test 1 = 10 psig ~4.2 slpm Test 3 = 30 psig ~4.2 slpm All for 20 min minimum
Operator: Dan Rinehart
Test Engineer: LK Jagoda

Test No.	Time	Chiller Temp.	Slurry Temp.	Slurry loop Flowrate	Filter Outlet Pressure	Permeate Pressure	Filter Inlet Pressure	Filtrate Flowrate Volume	Filtrate Flowrate Time	Filtrate Flowrate Flowrate	Tank Level	Comments
2	3:23	22.2	21.3	4.17	18.0	~2	19.0					Rotameter = 30
2	3:33	24.7	24.2	4.21	17.5	~2.5	19.0					R = 32
2	3:40	26.0	25.6	4.21	17.5	~2	19.0					R = ~32
2	3:48	24.7	23.2	4.19	17.0	~2	19.0					R = 32
2	4:00	22.1	21.0	4.17	17.0	~2	19.0					R = 30
2	4:14	25.4	24.9	4.20	18.0	~2	19.0					R = 32
1	5:04	24.1	23.6	4.00	8.0	~2	10.0					R = 14
1	5:08	22.0	20.3	3.98	8.0	~2	10.0					R = 12.5
1	5:11	20.9	19.4	3.99	8.0	~2	10.0					R = 12.0
1	5:16	20.9	19.7	3.98	8.0	~2	10.0					R = 12.0
1	5:20	21.4	20.3	3.98	8.0	~2	10.0					R = 12.5
1	5:24	22.1	21.2	3.99	8.0	2	10.0					R = 12.5
3	5:31	24.4	24.1	4.02	27.0	5	30.0					R = 50
3	5:43	24.9	23.8	4.2	25.0	4	28.0					R = 45
3	5:46	23.8	22.4	4.1	29.0	4	30.0					R = 49
3	5:56	23.2	22.3	4.07	29.0	4	30.5					R = 50

Data Sheet 1: Operating Data

Date: July 27, 1999
Tank Number: AN-107 Archive Sr/TRU ppt
Filter: 0.1 μm Mott-L (2nd)
Test Conditions:
Operator: KP Brooks
Test Engineer: R Lettau

Test No.	Time	Chiller Temp.	Slurry Temp.	Slurry loop Flowrate	Filter Outlet Pressure	Permeate Pressure	Filter Inlet Pressure	Filtrate Flowrate Volume	Filtrate Flowrate Time	Filtrate Flowrate Flowrate	Tank Level	Comments
1	10:01	29.4	28.4	3.9	51	2	54	40	7.44			Rotameter = 10
1	10:07	25.3	23.8	3.8	52	2	56	40	14.36			Rotameter = 6
1	10:11	23.6	21.9	3.85	52	2	56	30	12.83			Rotameter = 5
1	10:22	25.5	25.1	3.9	51	2	54	30	18.13			Rotameter = 4
1	10:31	25.9	24.7	3.8	51	1	54	30	12.09			Rotameter = 3-4
1	10:49	29.2	27.9	3.8	48	2	51	30	23.32			
1	11:02	off	23.9	3.9	53		57	30	28.12			Rotameter = 2
	11:09	Backpulsed twice at 70 psi										
2	11:11	31.4	32.1	4.1	37	1	39.5	30	14.72			Rotameter = 4
2	11:22	21.5	19.6	4.0	37	1	40	30	31.38			Rotameter = 2
2	11:31	21.1	22.1	4.0	37	1	40	30	33.34			
2	11:41	26.0	25.3	4.0	36	1	39	30	30.93			Rotameter = 1
2	11:54	22.3	20.4	4.0	37	1	40.5	20	25.44			
2	12:05	23.0	23.7	4.0	37	1	40	20	25.97			
	12:08	Backpulsed twice at 70 psi										
3	12:14	26.6	24.5	2.9	68	1	70	30	14.56			Rotameter = 4
	12:28	Backpulsed 5 times at 70 psi										
3	12:29	22.6	17.9	2.7	72	1	74	30	16.28			Rotameter = 4
3	12:39	26.8	23.4	2.7	71	1	72	30	34.60			
3	12:52	25.4	22.1	2.9	68	1	70	30	10.25			

Data Sheet 1: Operating Data

Date: July 27, 1999
Tank Number: AN-104 Archive Sr/Tru ppt
Filter: 0.1 μm Mott-L (2nd)
Test Conditions:
Operator: R Lettau
Test Engineer: LK Sagode

Test No.	Time	Chiller Temp.	Slurry Temp.	Slurry loop Flowrate	Filter Outlet Pressure	Permeate Pressure	Filter Inlet Pressure	Filtrate Flowrate Volume	Filtrate Flowrate Time	Filtrate Flowrate Flowrate	Tank Level	Comments
3	12:58	23.0	18.6	2.8	67	1	69	30	38.57			
3	1:07	23.2	19.5	2.8	68	1	70	30	42.50			
3	1:17	27.1	23.9	2.8	71	1	73	30	42.59			pressure jumped fluctuating as pump pulses / see LR3
3	1:28	23.7	19.0	2.9	103	1	107	30	54.97			
4	2:04	27.6	25.9	3.13-3.16	50	1	52	30	22.03			R=4
4	2:08	27.2	24.5	~3.1	~51-52	1	55	30	34.53			R=0
4	2:22	21.9	18.2	~3.1	67	1	60	30	46.62			readjusting pressure
4	2:35	25.9	23.5	3.2	53	1	56	30	49.72			
4	2:48	23.7	19.9	3.13	53	1	56	30	58.38			
4	2:59	24.5	21.8	3.11	53	1	55	30	52.71			
4	3:09	26.2	22.9	3.23	52	#1	55	30	56.60			
5	3:30	24.2	21.9	3.3	52	1	55	30	25.13			R=4
5	3:32	"	"	"	"	"	"	30	33.19			R=0
5	3:42	25.8	22.7	3.4	49	1	52	30	44.50			adj back to 55 psig
5	4:02	21.7	19.5	2.22	55	1	55					R=4-5
5	4:05	"	"	"	"	~1	"	30	39.78			
5	4:15	25.5	23.9	2.24	51	1	53	30	55.22			adj to 55 psig
5	4:30	22.1	19.5	2.38	53	1	55	30	67.38			adj flow back to ~2.25
5	4:42	24.9	23.1	2.25	54	1	56	30	64.62			
5	4:55	24.1	21.0	2.22	53	1	55	30	69.84			

→ Can't reach target flow !!
→ Restart - New target - 2.25 gpm @ 55 gpm

Data Sheet 1: Operating Data

Date: July 27, 1999
Tank Number: AN-~~101~~ Archive
Filter: 0.1 μm Mott-L (2nd
Test Conditions:
Operator: R. L. Rethau
Test Engineer: L.K. Jagoda

Test No.	Time	Chiller Temp.	Slurry Temp.	Slurry loop Flowrate	Filter Outlet Pressure	Permeate Pressure	Filter Inlet Pressure	Filtrate Flowrate Volume	Filtrate Flowrate Time	Filtrate Flowrate Flowrate	Tank Level	Comments
5	5:05	22.1	19.0	2.24	52	1	55	30	75.10			
6	5:23	27.4	25.5	~3.4	53	1	55					R=4
6	5:24	27.4	25.5	~3.4	53	1	55	30	28.47			R≈1
6	5:33	26.2	22.4	~3.46	52	1	55	30	45.87			
6	5:43	23.2	18.6	~3.45	53	1	55	30	52.16			
6	5:52	24.4	20.9	~3.5	52	1	55	30	50.25			
6	6:05	25.1	20.9	~3.5	52	1	55	30	54.22			
6	6:19	22.9	18.7	~3.5	52	1	55	30	58.72			
6	6:25	24.9	21.6	~3.5	52	1	55	30	56.31			

50 7/15/99

Hot Cutt

9:00pm - Blank filter is in running ~20psig

9:30 0.05 μ filter open (V20 & V2)
~15psig

V22 & V23 valve labels on old filter -

9:40 Bagged up old Matt filter to keep cleaner.

9:55 pm - Shut down

7/16/99

8:55 am Turned on the pump at ~5gpm at 15psi
Running pump thru filter cartridge with blank filter attached.

Pulled off water sample sent thru filter. (0.2um) (dead-end filter). W/ 10" Hg on outside vacuum gauge, filtered very slowly. Drained water - no particulate seen. Filled up a second time. Again filtered slowly at first & then stopped. No obvious solids on filter.

Vacuum system was down. Filters quickly and no obvious solids after 3 times.

9:50 Turned off system. Filtered material from V9 2 times in 150 mL filter. No obvious solids on filter.

Replaced 0.1 um Matt w/ new matt filter

Project No. 9953 Date of Work 7/15 & 7/16/99

Entered By [signature] Date 7/15/99

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Signed 1. _____ Date _____

2. _____ Date _____

13:03 $P_{in} = 20$ $T = 23.5$ $P_{out} = 2$ psi

Rotameter = 30

13:08 Rotameter = 26

13:16 Rotameter = 24

13:23 Rotameter = 25 ~4 gpm $P_{in} = 18$

13:32 Rotameter = ~~25~~ 27 $P_{out} = 2$ $P_{in} = 18$

$T = 26.7°C$

2:06pm - Finish test 2 (30 psig ~4.28 gpm) for 20 min.

Overall Ran 1 hr with very little drop in flux!!

Back pulsed 5 times.

2:10 Starting test #1 (10 psig ~4.2 gpm)

2:33 finished test #1

Back pulsing 5 times

2:37 Starting test #3 (30 psig ~4.28 gpm)

3:00pm finished test #3 - in 20 min dropped from 45=R 37=R

3:20 Had to tighten a valve - done -

Project No. 29983 Date of Work 7/16/99

Entered By [signature] Date 7/16/99

Disclosed To and Understood By

Signed 1. _____ Date _____

2. _____ Date _____

~ 4:30 pm AN-107 Shaken and poured into CUF

~4:45 Started system

Can't get pressure above 12 psig even with V1 mostly closed. -

Identified 2 problems

* Problem with V1 opening & closing - to retighten it.
* Building Air is low 60 psig max

5:20 - Played with V1 until can reach 70 psig on system.

5:26 Turn system to setup Condition one
F = 4.2 gpm
P = 55 psig

5:28 open V4

T = 0 R = 10
T = 0:49 R = 0
(49 sec)

Switch to glass flowmeter
Close V6

10 ml = 35 sec
20 ml = 1:20
30 ml = 3:09

Closed V4 = Collected Sample CUF AAN107-0 (filtrate)

* Attempted to backpulse / mostly with Air - couldn't fill backpulse chamber

Project No. 29953 Date of Work 7/14/99

Entered By [signature] Date 7/16/99

Disclosed To and Understood By

Signed 1. _____ Date _____

2. _____ Date _____

7/16/99

Set conditions to 4.2 gpm & 70 psi

#140 pm 5 ml / 51 seconds

V4 closed

Drained system - recovered almost all volume, lost 119.56 g.

[Probably left much of that lost weight in the filter as clogging solids]

8:05 - 1 L of 2 M NaOH has been pumping around about 1 hr. - Tried to back pulse - 1/4 L success - going to add H_2O from top & try again.

2 back pulses from the top - flux is much better!!

Refilling and back pulsing normally -
2 more back pulses -
opened V4 - R=10 - washed out R & motor & glass flow meter for ~30 sec

closed V4 - Back pulsing 6 more times

8:30 Draining

9:00 Back pulsed in through top 5 times - added another ~.5 L

9:12 running -

9:30 draining

9:35 refilling - (3 from top - back pulse)

9:40 running - 9:50 draining

9:55 refilling to leave for weekend

Project No. 1995 Date of Work 7/16/99

Entered By [signature] Date 7/16/99

Disclosed To and Understood By

Signed 1. _____ Date _____

7/20/99

12:40 AM - System up & running -

Visual inspection pre-start up showed water visually clear, after running ~ 15 min still clear.

Plan - take pH - hook up .05 μ filter to remove any remaining solids

1:20 pH ~ ≤ 8.0

1:40 Drained - partially and put ~ 1.5 liter of fresh H_2O into tank

1:55 Running through 0.05 μ filter ~25 psig ~5.7 gpm

2:05 Backed off flowrate to ~5.2 gpm -

2:05 Chiller on T ~ 38C when turned on
2:08 → T dropped quickly to < 30 C

2:15 pulsed flow rate 4 times (trying to make sure that solids are knocked out of fittings)

2:48 pulsed flow rate 5 times

3:04 V10 closed (0.05 μ off)

3:06 Conditions set to 20 psig ~ 4. gpm
3:12 → 19 psig - 4.15 gpm

T = 0	R = 30	T = 30	R = 33
T = 5	R = 30	T = 40	R = 31
T = 13	R = 30	T = 50	R = 30
T = 20	R = 32	T = 61	R = 32

Project No. 29983 Date of Work 7/23/99

Entered By [signature] Date 7/23/99

Disclosed To and Understood By

Signed 1. [signature] Date

2. Date

AN-107 Hot Cuff Clean-up & water testing

7/23/99

4:20 Back pulsed 4 times -

4:20 New conditions – Test #1 –
~4.0 gpm & 10 psig.

T = 0 R = 12

4:25 Rubber hose from glass flowmeter came off -
Stopped system

4:45 Fixed glass flowmeter connector -
added ~1.0 L DI H_2O

5:00pm Set conditions to 10 psig ~4.0 gpm

T = 0 R = 14
T = 5 R = 12.5
T = 14 R = 12.0
T = 20 R = 12.5

5:25 Finished – Back pulsing 3 times

New Conditions 4.2 gpm, 30 psig

T = 0 R = 45
dropped p to 27 psig -
adjusted to 30
T = 1 R = 50

* Water level too low for flow rates - turning to foam - R dropped to < 30 Fluid
Added ~1 more Liter (~¾ up of site glass on tank)
~~T = 12~~ Clean it up - R went back to 45
T = 14 R = 45 47 P = 27-28
adj pressure back to 30 R = 49,
T = 20 R = 49
T = 25 R = 50

Data from H2O test
Recirculation temp CUFF 2
(Temp full @ ~5:45 pm)

Project No: 29953 Date of Work 7/23/99

Entered By [signature] Date 7/23/99

Disclosed To and Understood By

Clean

7/26/99 Compared 18" TC to Thermometer temp @ a [illegible] at ~ 40~50°C as a check of the calibration. All measurements were within 0.3°C of each other in a stirred beaker of water.

18" TC = 54.0°C
Thermometer = 53.5°C
6" TC = 53.8°C

18" TC = 54°C
Thermometer = 53.7°C

7/26/99 Tare wts using Mettler AT400
384-06-Q-004

Sr/TRU ppt	75 mL (full)	184.3352 g
1M $NaMnO_4$	50 mL (full)	112.1823 g

TCIX-01	16.8953 g
TCIX-02	17.0301 g
Mn-21	17.0031 g
Mn-22	16.9183 g
Mn-23	16.8969 g
Mn-24	16.9147 g

1 liter container From AN-107 Archive - 2 samples removed ~ 20mL

TCIX-01	43.654 g - full weight	(using cell PM400-cal → 43.650 g (recheck on 7/27/99)
MN-21	41.325 g - full weight	
	41.319 g (recheck 7/27/99)	

13:39 Began adding $Sr(NO_3)_2$ Solution to AN-107.
T = 34.1°C
Some of the Sr solution is lost due to difficulty pouring out of the container (a few drops)

13:45 Finished the addition of the Sr solution.
Stirred for 10 minutes after Sr Addition complete
Turned stirrer off to video-

13:57 Turned off video time stamp

14:04 Turned stirrer back on

Project No. 29953 Date of Work 7/26/99

Entered By ~~T. Brooks~~ Date 7/26/99

Disclosed To and Understood By

Signed 1. _____ Date _____

2. _____ Date _____

Sr/TRU ppt

14:13 Add $NaMnO_4$ solution
T = 33.6°C
Bottles are spilling significantly - they are hard to pour. Possibly ~2 mL lost to side of container.

14:17 Addition is complete

14:19 Turned off stirrer. The stir plate has quite a lot of purple MnO_4^-.

14:29 Turned on heat T = 38°C

14:52 Reached 50°C

15:37 T = 53.2

16:58 - took video of container

18:55 - heater off.

* Note over last 3 hrs T = 50.4 -> 51.8 C @ lowest setting on hot plate temperature ~~rises~~ climbs very slowly. About every 30 min ~~shut~~ (as temp approached 52 C) the hot plate was turned off (stirrer left on) for ~10 min until the temp dropped back to almost 50 C

19:21 T = 48.2

19:29 T = 47.4 Scanner T.C. DAS off - Still mixing well. - Removing T.C so lid sits tighter to reduce evaporation.

1/27/99 T = 35.4°C Put T.C. back in

8:18 Took short video

8:20 Turned off stirrer to allow settling. Took external filter off.

8:55 Took 2 samples from top of reaction vessel TcIX-02 & Mn-22.

9:02 Weighed containers

Project No. 29953 Date of Work 7/27/99

Entered By [signature] Date 7/26/99

Disclosed To and Understood By

1 – 10:00 am started system Filtrate flux [illegible]

10:06 am took Sample Mn-23 Filtrate

10:30 am took sample Mn-24 Filtrate – #1

Weights of full samples

Tc IX-7	41.345 g
Mn-22	40.943 g
Mn-23	31.553 g
Mn-24	30.440 g

2 – 11:10 am started new condition

11:40 am took sample Mn-25 Filtrate # 2

Mn-25 32.508 g (Full sample wt)

Filtrate #3

12:55 Mn-26 31.399 g (Full sample wt.)

1:28 For the last 30-40 min the pump has been making a throbing sound, the pressure has fluxuated with the pump pulses. It has also been drifting up to 75 psi down to 67. Ralph has adjusted it several times.

* Tare wts taken in cell for Bottles Mn-27-30

Mn-27 = 16.903
Mn-28 = 17.098
Mn-29 = 16.950
Mn-30 = 16.961

Test condition 3 ended @ 1:29 - Backpulsing 5 times (from V10)

1:50pm - Taking 2 slurry samples

Sample Mn-27 = 28.845 (Sample + Slurry)

* After dumping first pull back into tank there was some residue in the vial which became part of sample.

Slurry #1

Project No. 29953 Date of Work 7/27/99

Entered By K Brooks Date 7/27/99

Disclosed To and Understood By

Signed 1. _____ Date _____

2. _____ Date _____

AN-107 Archive testing

Sample Mn-28 = 28.708 g (Sample bottle + slurry)
↳ Slurry #2

14:01 (2:01 pm) Starting test 4 - 55 psig 3.13 - 3.16 gpm

~14:30 Flushed sampling system

14:50 Took filtrate sample - Mn-29 = 27.037 g (sample bottle)
↳ Filtrate #4

1511 Finished Condition 4

Back pulsing ~~4~~ times

1530 Starting Condition 5 testing - ~~target~~ 5.23 gpm / 55 psig
Max flow 3.3 - 3.4 gpm 55 psig.
Air pressure is to low to achive high flow rates & Having bubbling Air. chaled -

1601 - Re starting Condition 5 with a new target of 2.25 gpm & 55 psig.

Back pulsed 3 times before start

Putting in two more sample bottles
Mn 31 - tare = 16.8608
Mn 32 - tare = 16.9707

4:44 Sample Mn-30 = 33.347 g (Sample bottle & slurry)
↳ Filtrate # 5

~~AM~~ 5:08 Finish Condition 5 - Back pulsing 3 times

5:23 Start Condition 6 - ~3.5 gpm / 55 psig

Sample Mn-31 - ~~tare~~ 27.244 g (sample bottle & slurry)
↳ Filtrate # 6

6:25 Finished Condition 6

Project No. 2995-3 Date of Work 7/27/99

Entered By [signature] Date 7/27/99

Disclosed To and Understood By

~~System Clean Up~~

7:05pm Drained - 850 ml of AN-109 Archive Recovered

7:30 Added 1 L .2 M NaOH pumped around ~15 min. While pumping allowed to go through filtrate System ~ 5 min - open and closed V6 3 times to rinse glass flow meter (filled to top of overflow) Then Back pulsed 3 times.

7:53 Draining - Collecting into bottle labeled 1st Rinse AN-109

8:14 Refilled with 1.5 L H_2O, Running @ ~50 psig ~ 4.5 gpm - trying to rinse tank walls with the same spatter pattern that dirtied them,

8:25 Drained -

Refilled with 2 L DI H_2O - Ran 15 min.

9:05pm Shut down

8/99 8:00am Turned on CUF at 30 psi & 4.5 gpm

8:30 am Backpulsed & drained

8:49 am Added liquid w/ tube to backpulse chamber Performed 5 times

9:50 am Added 1.5 l DI water into system and ran around for a while at 30 psi & 5 gpm Rinsed filtrate sample line & upper loop

10:30 Drained -

Project No. 29958 Date of Work 7/27/99

Entered By [signature] Date 7/27/99

Disclosed To and Understood By

Signed 1. _____ Date _____

2. _____ Date _____

AN-107 Archive – Composite prep.

7/28/99

Preparation of Composite Mn-32 (tare 16.97

			total wt Sample only	# g of indiv. Δ wt. =
11:31am	tared	—	0.00 g	
	After ~2 ml	Mn-24	2.377 g total	2.377 g
	After ~2 ml	Mn-25	4.789 g total	2.412 g
	After ~2 ml	Mn-26	~~6.518~~ 7.192 g total	2.403 g
	After ~2 ml	Mn-29	9.543 g total	2.351 g
	After ~2 ml	Mn-30	12.076 g total	2.533 g
	After ~2 ml	Mn-31	14.418 g total	2.342 g
			check ✓	14.418 g

Total wt. Bottle Plus composite sample = 31.386g

~1-2:30 Tranfed samples out to go to SAL for an

Samples transfed - TCTx-01 Mn-32 Mn 23
TCTx-02 Mn 21 Mn 28
Mn-31 Mn-22 Mn 27

2:45 Refilled with ~1.5L H_2O -
ran ~30 min

3:20 Brushed down sides - water now foamy & bro
again

3:40 Took plt - ≥7,5 or less - ∴ Can attach fil
now.

4:00 Shut down until tomorrow

Project No. 29953 Date of Work 7/28/99

Entered By [signature] Date 7/28/99

Disclosed To and Understood By

Signed 1. _____ Date _____

Archive

199

7:50am - up and running to remix, any solids in system. - resuspend,

10:19 Hooked up & running 0.05m filter system, 25psig ; ~4.8 gpm - Very Foamy & Cloudy

10:55 Back pulsing with Clean H_2O from the top. (Twice)

12:00 Cleaned until 12:00 still a little cloudy

1:45 Continued to clean until 1:45 - Crystal clear visually - V2 & V10 closed.

1:55 - Testing - 20 psig / 4.2 gpm
T=0 R=20-21
T=8.5 R=17

2:05 V4 off - Back pulsing 10 times

2:29 Cleaning again - V2 & V10 open to 0.05m filter

2:50 Pulsed flow 6-7 times to ~~create~~ turbulence/different flow patterns.

3:15 - Partially drained (put filter discharge into waste container) Refilled with 2L all added from the top as Clean H_2O back pulses.

3:45 - Running again - H_2O brownish & cloudy.

4:23 - building air down - pump shut off

Project No. 29953 Date of Work 7/29/99

Entered By [signature] Date 7/29/99

Disclosed To and Understood By

Signed 1. _____ Date _____

_____ Date _____

AN-107 clean up

7/30/99

7:50 System up & running - V2 & V10 open ∴ filtering through 0.05 M filter,

8:55 looks clear visually - V2 & V10 closed -

Set conditions to 20 psig ~4.2 gpm
Open V4 - re adjust to 20/4.2 gpm

T = 0	R = 22.5
T = 6	R = 20
T = 10	R = 20
T = 14	R = 20
T = 17	R = 18
T = 20	R = 17.5

9:23 - Testing shows system is still not clean - Going to add acid.

9:24 - Back pulsing ~~5~~ (five) times then draining

9:41 - Back pulsed 3 times Fresh H2O from top

10:17 looks clean - doing a complete drain

10:39 Pouring Acid in - through top. (1L 1M HNO_3 0.2 M citric

11:25 running acid - 25 psig - 4.1 gpm (pulsed a few times

12:46 - Rinse Acid finished - very foamy -

12:48 Draining - Acid greenish now

1:20 Refilled Clean H2O - running (30 psig ~ 4.5 gpm)
Open V4 3 times - R pegs @ 60+/gpm fills fast.
Back pulse 5 times

Project No. 29953 Date of Work 7/30/99

Entered By [signature] Date 7/30/99

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1:34 Draining.

1:40 * Note - Leak detected in the Back pulse Chamber ~~near~~ @ bottom of Site glass.

2:00 Refilled and running @ 30 psig ~4.

2:13 Stopped / Draining /

2:20 Refilled - Ran -

2:45 Drained
pH = ~3 - 3.5

2:55 Refilled Running pH = 3.5 - 4.0

3:20 Dumped -
3:30 Refilled

3:46 pH = 4.5 !!

Shutting down -

Project No. 29953 Date of Work 7/30/99

Entered By [signature] Date 7/30/99

Disclosed To and Understood By

Signed 1. _____ Date _____

2. _____ Date _____

Appendix B: Test Instruction-063 and Log Book Entries

Sr/TRU Removal from Archived AN-107 with Minimal Reagent Addition

Log Book Entries

PNNL Test Instruction

Document No.: BNFL-TI-29953-063
Rev. No.: 0

Title: Sr/TRU Removal from Archived AN-107 (previously treated by Cs IX) with Minimal Reagent Addition

Work Location: RPL SFO HLRF

Page 1 of 8

Author: Richard T. Hallen

Effective Date: Upon Final Approval
Supercedes Date: New

Use Category Identification: Information

Identified Hazards:
___Radiological
__ Hazardous Materials
__ Physical Hazards
__ Hazardous Environment
__ Other:

Required Reviewers:
__X__ Technical Reviewer ____ SFO Manager

Are One-Time Modifications Allowed to this Test Instruction? __X__ Yes __ No

NOTE: If Yes, then modifications are not anticipated to impact safety. For documentation requirements of a modification see SBMS or the controlling Project QA Plan as appropriate.

On-The Job Training Required? ____ Yes or __X__ No

FOR REVISIONS:
Is retraining to this procedure required? ____Yes __X__ No

Does the OJT package associated with this procedure require revision to reflect procedure changes?
____Yes ____No __X__ N/A

Approval	Signature	Date
Author	Richard T. Hallen	10/25/99
Technical Reviewer	V T Brooks	10/25/99

Controlled Document

1.0 Applicability

This test instruction is to be used to perform studies on permanganate treatment for Sr/TRU removal from archived AN-107 waste that has been previously treated by cesium ion exchange resin and prepare addition treated waste for sulfate removal studies. The precipitation will be conducted in SAL and solids removed by deadend filtration. Approximately 1-L of AN-107 waste (use C3E4 first then C3E5, and/or C3E6) will be used.

2.0 Supporting Documents

This test instruction is a stand-alone document. Background information on permanganate treatment can be found in PNNL Test Plan BNFL-TP-29953-013, which contains an overall description of the project, ES&H compliance, emergency response, and the hazards assessment and mitigation. Past scoping tests on the permanganate treatment of AN-107(cesium removed) are described in detail in test instructions, TI-29953-037, -040, -041, and -043.

3.0 Responsible Staff

The staff responsible for executing this test plan are as follows.

- Task Manager – Dean Kurath
- SFO Manager – Rick Steele
- Test Scientists/Engineers – Kriston Brooks and Rich Hallen
- Hot Cell Technician – Vaughn Hoopes
- Radiological Control Technician

4.0 Materials, Equipment, Supplies and Reagents Needed

4.1 Materials Required

1. Four 20 mL glass scintillation vials for samples, pre-labeled on top and side as follows: MR-01 - MR-04.
2. One 250 mL filtrate Storage Bottle.
3. 1-L size disposable filtration unit, 0.45 um filter.
4. 150-mL disposable filtration unit, 0.45 um filter.
5. One 150 mL breaker or bottle for transferring and weighing the Archived AN-107
   250 ml Plastic beaker T= 14.7621

4.2 Equipment

1. 160 gram balance (largest available in SAL)
2. Calculator
3. 2-L erlenmeyer flask
4. hot-stir plate
5. Thermometer or thermocouple/reader
6. big stir bar
7. watch
8. Catch pan to use as secondary containment.

4.3 Reagents Needed

✓1. 48 mL of 19M (50 wt %) NaOH
✓2. 57 mL of 1.0M Sr(NO3)2
✓3. 34.2 mL of 1.0M NaMnO4
✓4. 1 liter of Archive AN-107 - #4 AN107-C3E-4
& #5 AN107-C3E-5

4.4 Other Supplies

✓1. Control Copy of this Test Instruction
✓2. Laboratory Record Book (record LRB# 13733)

5.0 Test Instructions for Permanganate Treatment

The laboratory record book (LRB) shall be used to record other testing information as required by this procedure and all test conditions not stated by this procedure.

Cross-contamination between samples and contamination of samples from outside sources must be minimized at each step. Use new supplies and bottles for each sample as much as possible.

Keep all test materials in sealed containers as much as possible to prevent them from drying.

5.1 Pre-start

5.1.1 Inventory materials, equipment, supplies, and reagents to ensure all required items are available. Assure that all materials have been modified for remote handling. (The large Erlenmeyer flask will need to be banded so the waste can be poured out. The 1-liter filter flask is large and hard to handle in the cell, band the flask.)

5.1.2 Do the following and initial and date when completed.

AG 10/26/09 Review the work instructions in this TI.

5.1.3 Record the weights of all the materials/supplies before they are transferred into the hot cell.

	ID#	Empty	Full
Sample vials	MR-01	16.8154 g	42.9240 g
	MR-02	16.8161 g	~~41.7626~~ g 41.9686 g
	MR-03	17.0376 g	43.9364 g
	MR-04	16.8920 g	_____ g

Stir Bar 38.4447 g Erlenmeyer Flask 736 g

Large
Filter unit receiver 571 g cap 15.2208 g
Filter unit 85.4503 g lid 18.8515 g

Small Tare
Filter unit receiver 39.4161 g cap 11.1947 g
Filter unit 30.4118 g lid 8.3338 g

Filtrate storage bottle 587 g w/lid

5.2 Start-Up

5.2.1 Obtain the following information:

M&TE List:

Balance #1a - (SAL outside of cell)(160g)
360-06-01-040 Exp 2/00
Balance #2a (SAL outside of cell) (Readability to .001g)
360-06-01-043 Exp 8/2000

_____ Balance 1:

Calib ID 360-06-01-016 Calib Exp Date 2/00

Location SAL Cell #2

Thermocouple or Thermometer: TC-325-415

Calib ID ~~TC-325-405~~ 416 Calib Exp Date ~~8-00~~ 1-2001

Location Cell 3 Type K

5.2.2 Record the identification number on the bottle of archived AN-107 (verify/identify sample is AN-107 id#).

Bottle Label(s) AN-107-C3E-4
AN-107-C3E-5

5.3 Sr/TRU Removal by Permanganate Treatment

5.3.1 Setup the ppt reaction equipment as specified. The precipitation is to be conducted in a 2L flask. Secondary containment should be used to allow recovery from a possible breach of the 2 L flask. Place stir bar in flask.

Add 1000 mL of archived AN-107 to the Erlenmeyer Flask. Because the balance will only measure 160 grams, 10 transfers should be completed, each with approximately 100 mL of waste.

Tare weight bottle	**Add ~100mL**	**After Waste Pour in Flask**	
AN-107 C3E-4 14.7621 g	138.8350 g	14.9370 g	transfer 1 = 123.8980 g
now add another 100 mL*	132.3573 g	14.9057 g	transfer 2 = 117.4516 g
now add another 100 mL	125.9800 g	15.0023 g	transfer 3 = 110.9777 g
now add another 100 mL	129.9138 g	14.9302 g	transfer 4 = 114.9836 g
now add another 100 mL	131.0341 g	15.0090 g	transfer 5 = 116.0251 g
now add another 100 mL	123.1172 g	14.9664 g	transfer 6 = 108.1508 g
AN-107 C3E-5 now add another 100 mL	131.3619 g	15.0333 g	transfer 7 = 116.3289 g
now add another 100 mL	129.4123 g	14.9678 g	transfer 8 = 114.4445 g
now add another 100 mL	137.0690 g	14.9162 g	transfer 9 = 122.1528 g
now add another 100 mL	131.0906 g	15.0100 g	transfer 10 = 116.0806 g
			Total = 1,160.8936 g

* on the 8th addition more sample was added to the cup before weighing so an avg. of the other nine wts was used.

Density = _____g/mL Total volume = Total mass/density = _____ mL

Turn on stirrer. Turn on the temperature recording devise. Record temperatures of cell and waste. Cell Temperature 25°C Waste Temperature 31.5°C @ 8:30am RJS 10/25/99

→ Remove 50mL of waste for sampling/filter below - record weight removed

5.3.2 While continuously stirring the waste, personnel are to slowly (over a 2 minute period) add 48 mL of 19M (50 wt %) NaOH. This is the entire content of the bottle labeled 19M NaOH.

Full
Tare bottle of 19M NaOH 98.0334 (wt outside of cell) 6:53 Added over 5 minutes
Tare Empty bottle 25.7361
Weight Added 72.2973 g

5.3.3 While continuously stirring the waste, personnel are to slowly (over a 5 minute period) add 57 mL of 1.0 M $Sr(NO_3)_2$. This is the entire content of the bottle labeled 1M Sr.

Full
Tare bottle of 1M Sr 91.3135 (wt. outside of cell) 8:12 – 8:18 am
Tare Empty bottle 22.2641 added Sr
Weight Added 69.0494 g

5.3.4 While stirring, slowly (over a 5 minute period) add 34.2 mL of 1 M $NaMnO_4$. This is the entire content of the bottle labeled 1M NaMnO4

Tare bottle of 1M NaMnO4 97.2778 (wt. outside of cell) 7:27 start
Tare Empty bottle 57.9487 7:33 complete
Weight Added 39.3291 g

5.3.5 Allow the waste to thoroughly mix after addition of all of the reagents, i.e. stir for 30 minutes. Began heating 8:22 am

5.3.6 Heat the waste mixture at 50 ± 5°C with stirring for 4 hours. Record temperature. RJS 10/25/99

Started 4 hrs 8:33 am – 45°C collect above-50mL

→ While the waste is digesting for 4 hours, transfer 20-mL of waste ~~from the original bottle~~ to vial MR-01 and record the weight of sample. This sample will be the unfiltered control, and is to be digested with acid as is for analytical. Duplicate samples need to be digested and submitted for analyses. Then filter approximately 30 mL of the original waste with 0.45 um filter. Use the filtrate to determine the density of the Archived AN-107 in duplicate using volumetric flasks (use 10 mL ball flask)

(1)
Tare flask 14.2162
Flask plus 10 mL of waste 26.8939
Weight of 10 mL of waste 12.6777 density of waste 1.268 g/mL

(2)
Tare flask 9.3997
Flask plus 10 mL of waste 22.0781

16.8159 g

Weight of 10 mL of waste 12.6784 density of waste 1.268 g/mL

Transfer 20 mL of the filtrate to vial MR-02 and record the weight. This sample is the filtered control. Sample in duplicate, acid digest, and submit for analyses.

12:41 pm - stirrer and heater off

5.3.7 After 4 hours of digesting, turn off the stirrer and allow the waste to cool to 25 ± 5°C. If possible, use a video recorder to document the settling behavior of the waste mixture. Record the volume of settled solids if possible. — mL See video - maybe 100 mL clear supernate

5.4 Solids Removal with the Deadend Filter Unit

5.4.1 Tare the 1-L receiver bottle/lid and filter assembly/lid of the filtration unit (preferably before it is sent into the cell). Make sure receiver bottle is banded for remote handling.

Tare of receiver bottle — and lid 586 g
Tare of the filter assembly 104.2824 (with lid) and lid —

see section 5.1 this instruction.

5.4.2 Assemble 1-L filtration unit and filter all of treated waste in the erlenmeyer flask Some solids should have settled on cooling/setting. Decant most of the supernate from the flask to the filter. The supernate should filter faster without all of the solids/cake on the filter unit. When approximately half the material has been filtered, swirl the bottle to suspend the solids. Continue filtering the slurry. If excessive solids remain in the bottom of the bottle, small amounts of filtrate can be used to rinse the solids from the bottle. Record weight of empty flask if possible. Filter the solids until compacted on filter and no free liquid remains. Disassemble filter unit and weigh. If necessary to get filtrate to Sandy Fiskum by COB Wednesday, stop filtration and transfer 250 mL of waste to a tared, 250 mL storage bottle.

~700 mL

Done last
Tare of receiver bottle/lid and filtrate 1438.4 g Weight of filtrate 587 g tare = 851.4
Tare of the filter assembly/lid and wet solids 199.5 g Weight of wet solids 952.176 g + ~~tare~~ stir bar
Tare = 104.2824 g = 567.729 g

5.4.3 Transfer 20-mL of filtrate from the filter flask to vial MR-03. Determine the density of the filtrate in duplicate using volumetric flasks (use at least 10 mL ball flask, prefer 25 mL.)

Tare flask 19.9085 (3)
Flask plus 25 mL of filtrate 51.3483
Weight of 25 mL filtrate 31.4403 density of filtrate 1.2576 g/mL

Tare flask 20.3640 (4)
Flask plus 25 mL of filtrate 51.0622
Weight of 25 mL filtrate 31.4982 density of filtrate 1.2599 g/mL

Transfer the remaining treated, archived AN-107 filtrate to the sample storage bottle. Collect the solids on the filter and save for future work.

Tare of 250mL bottle - w/lid = 229.54
w/o lid = 214.95

5.5 Experimental Clean Up and Sample Disposition.

Clean up all of the equipment used.

Do not discard any samples without written instructions from the task manager. Duplicate samples were collected at all sampling points. The duplicates are to be retained until review of the analytical data is completed and notification is writing is received to dispose of these samples.

6.0 Sample Analysis

The point of contact for the sample analysis is Mike Urie and Rick Steele.

Prepare and submit all samples in duplicate. Analyze all samples for the following: Am/Eu by GEA, Sr-90 analysis, Tc-99 beta scintillation without oxidation, and Na by ICP-AES (report all detected analytes).

Table 1. Samples and Their Required Analyses

Process Variable	Vial ID	Sample Type	Sample Preparation	Analysis Description[a]
Archived AN-107	MR-01	as received	acid digest	Am/Eu/Sr/Tc, Na
Archived AN-107	MN-02	as received	0.45 um filter, acid digest	Am/Eu/Sr/Tc, Na
Treated AN-107	MN-03	filtrate	acid digest	Am/Eu/Sr/Tc, Na

(a) Descriptions of analyses are contained in Table 2.

Table 2. Description of Analyses

Constituent	Analysis Method	PNNL Procedure No.
Americium-241, Eu isotopes	GEA	PNL-ALO-450
Strontium-90 (Yttrium-90)	Separations and Beta Counting	PNL-ALO-476/431
Tc-99	Beta Counting without chemical oxidation	
Metal Ions (see Table 5 list)	ICP-AES	PNL-ALO-211/280

Table 3. Analytical Requirements for Supernate/Filtrate

Analyte	Supernate/Filtrate Minimum Reportable Quantity microCi/gm	Analysis Method
Americium-241	1E-03	GEA
Europium-154	1E-03	GEA
Europium-155	1E-03	GEA
Strontium-90	1E-01	Chemical Separation & Beta Count
Technetium-99	1E-02	Beta Scintillation without sample oxidation
	microgm/gm	
Al	NR	Acid Digestion followed by ICP-AES
Ba	NR	
Ca	NR	
Cd	NR	
Co	NR	
Cr	NR	
Cu	NR	
Eu	NR	
Fe	NR	
K	NR	
La	NR	
Mg	NR	
Mn	NR	
Mo	NR	
Na	2.3E+05	
Ni	NR	
Pb	NR	
Si	NR	
Sr	NR	
Ti	NR	
U	NR	
Zn	NR	

NR no minimum reportable quantity but report data for all analytes detected

Treatment of Archived AN-107 with minimal reagent additi double per Mike Johnson

to 500 ml of waste	19M NaOH	0.024 Liter	or	0.912 moles
	1M Sr	0.0285 Liter	or	0.057 moles
	1M MnO4	0.0171 Liter	or	0.0342 moles

Balance number: 380-06-01-013
Calibration date: 2/00

Makeup 50 % NaOH solution ~19 M
use NaOH pellets
40 grams/mole (FW) (lot # 01012EG) ACS reagent - 97+%

Tare 50 mL bottle 24.8879 grams
add 36.48 grams NaOH bottle + NaOH 61.4279 grams
add 36.48 g water 97.9663 grams
actual weight of NaOH added _____ grams
actual weight of water added _____ grams

Label bottle with ID, 50% NaOH total weight 98.0319 gram
48 mL

Makeup Sr(NO3)2 solution
use Sr(NO3)2
211.63 grams/mole (FW) (lot #

Tare 100 mL bottle 21.9174 grams
add 12.0629 grams Sr(NO3)2 bottle + Sr(NO3)2 33.9904 grams
add 57 g of water 91.3185 grams
actual weight of Sr(NO3)2 added _____ grams
actual weight of water added _____ grams

Label bottle with ID, 1M $Sr(NO_3)_2$ 57 mL 91.3191 grams

Makeup NaMnO4 solution
use NaMnO4.H2O
159.94 grams/mole (FW) (lot #

Tare 100 mL bottle 57.6883 grams
add 5.46995 grams NaMnO4 bottle + NaMnO4 63.1533 grams
add 34.217.1 g of wat 97.2223 grams
actual weight of KMnO4 added _____ grams
actual weight of water added _____ grams

Date prepared: 10/25/99
Prepared by: Richard L Hall
Work Package Number: W53400

1M NaMnO4 97.2792 g
34.2 mL

COPY

BNFL Inc.

River Protection Project
Waste Treatment Plant
3000 George Washington Way
Richland, WA 99352
Tel: (509) 371-3500
Fax: (509) 371-3504

Mr. Eugene Morrey
Pacific Northwest National Laboratory
P.O. Box 999, MSIN P7-28
Richland. Washington 99352

Direct tel: 509-376-1982
Direct fax: 509-376-7127

CCN #: 007525

CC: Kriston Brooks
Rich Hallen
Dean Kurath
Dave Blanchard
Sandra Fiskum

Dear Eugene:

Contract No. DE-AC06-96RL13308 – W375 – PREPARATION OF ADDITIONAL ARCHIVED AN-107 SOLUTION FOR SULFATE SEPARATION TESTS

References:

1. Document, BNFL-TI-29953-061, revision 0, "Sulfate Removal Studies from AN107 Archived Waste," Battelle Pacific Northwest Division, Richland, Washington, dated October 18, 1999.

2. CCN# 004960, M. E. Johnson, BNFL Inc. to E. V. Morrey, Battelle, "Conditions for Precipitation of Sr/TRU in Archived 241-AN-107 Sample," Battelle, dated July 28, 1999.

This letter specifies conditions for conducting Sr/TRU precipitation with ~500-ml of archived AN-107 solution that has previously been treated to separate cesium using crystalline silico-titanate. BNFL Inc. is requesting Battelle prepare the pretreated archived AN-107 solution for use in additional sulfate removal tests.

Battelle is conducting tests with the archived AN-107 solution to evaluate methods for separating sulfate from Envelope C waste solutions (ref. 1). The archived AN-107 solution had previously been processed through a column that contained crystalline silico-titanate to separate cesium. Battelle used a small portion of the archived AN-107 solution to demonstrate Sr/TRU separation using sodium permanganate and strontium nitrate. Battelle then used all available pretreated, archived AN-107 solution for sulfate separation tests. Battelle needs to conduct the Sr/TRU precipitate process with additional archived AN-107 solution.

Battelle personnel conducted Sr/TRU precipitation and filtration tests with the archived AN-107 solution using the following test conditions.

➢ Starting Conditions for the archived AN-107 solution

• Na:	5.68M	Density:	1.256 gm/ml
• Volume:	881.9 ml		
• Am-241:	0.174 μCi/gm	Co-60:	0.0616 μCi/gm
• Eu-154:	0.232 μCi/gm	Eu-155:	0.166 μCi/gm

CCN #: 007525

- Sr-90: 38.5 μCi/gm

➢ Reagents Concentrations and Conditions
- Free hydroxide (calculated) was 0.8M,
- Final $NaMnO_4$ molarity was 0.05M,
- Final $Sr(NO_3)_2$ molarity was 0.075M
- Heat mixture to 50°C for 4-hours, then cool and filter

➢ Final Conditions for the archived AN-107 solution before filtration

• Na:	4.83M	Density:	1.226 gm/ml
• Volume:	1014.53 ml	Dilution factor:	1.1247 (mass basis)
• Am-241:	0.00485 μCi/gm	Co-60:	0.0545 μCi/gm
• Eu-154:	0.0135 μCi/gm	Eu-155:	0.0097 μCi/gm
• Sr-90:	1.52 μCi/gm		

BNFL Inc. has selected Sr/TRU precipitation conditions different than those originally specified for the archived AN-107 sample in order to reduce the amounts of manganese hydroxide and strontium carbonate added to the high-level waste.

BNFL Inc. requests Battelle personnel to react ~500-ml of archived AN-107 solution with sodium hydroxide, 1M sodium permanganate, and 1M strontium nitrate solutions to achieve final added concentrations of ~0.8M OH (calculated), 0.03M (± 5%) MnO_4, and 0.05M (± 5%) Sr. Battelle personnel are to constantly mix the slurry, heat the slurry to 50°C for 4-hours, then cool and dead-end filter using a 0.45-μm filter. Battelle personnel are to sample and analyze the archived AN-107 supernate before and after reagent additions to determine the supernate density, concentrations of Na, Sr-90, Am-241, Eu-154, Eu-155, and Tc-99 by beta scintillation without sample oxidation. The minimum reportable quantities for Na, Sr-90, Am-241, Eu-154, Eu-155, and Tc-99 are 2.3E+05 μgm/gm, 0.1μCi/gm, 1E-03μCi/gm, 1E-03μCi/gm, 1E-03μCi/gm, and 0.01μCi/gm, respectively.

Battelle should accumulate costs for this activity as part of the sulfate removal task authorized by Mr. Bill Roberson to Ms. Whelan on October 14, 1999. A final charge control package should be submitted to BNFL Inc. for all sulfate removal tasks in November 1999.

Yours sincerely,

Michael E. Johnson
Pretreatment Technical Manager

MEJ/ctf

CCN #: 007525

cc:

Fittro, C.T. w/o	BNFL Inc.	A212
Slaathaug, E. w/o	BNFL Inc.	E213
Johnson, M.E. w/o	BNFL Inc.	B268
PDC w/o	BNFL Inc.	K110

T1-063

BNV-13733 Page 65

reagent prep for T1-063

eatment of Archived AN-107 with minimal reagent additi double per Mike Johnson

00 ml of waste 19M NaOH	0.024 Liter	or	0.912 moles
1M Sr	0.0285 Liter	or	0.057 moles
1M MnO4	0.0171 Liter	or	0.0342 moles

Balance number: 380-06-01-013
Calibration date: 2/00

lakeup 50 % NaOH solution ~19 M
se NaOH pellets
40 grams/mole (FW) (lot # 01012EG) ACS reagent - 97+%
are 50 mL bottle 24.8879 grams
dd 36.48 grams NaOH bottle + NaOH 61.4279 grams
add 36.48 g water 97.9663 grams
actual weight of NaOH added _____ grams
actual weight of water added _____ grams
abel bottle with ID, 50% NaOH total weight 98.0319 gram
48mL

lakeup Sr(NO3)2 solution
se Sr(NO3)2
211.63 grams/mole (FW) (lot #
e 100 mL bottle 21.9174 grams
dd 12.0629 grams Sr(NO3)2 bottle + Sr(NO3)2 33.9904 grams
add 57 g of water 91.3185 grams
actual weight of Sr(NO3)2 added _____ grams
actual weight of water added _____ grams
abel bottle with ID, 1M Sr(NO3)2 57 mL 91.3191 grams

lakeup NaMnO4 solution
se NaMnO4.H2O
159.94 grams/mole (FW) (lot #
are 100 mL bottle 57.6883 grams
dd 5.46995 grams NaMnO4 bottle + NaMnO4 63.1533 grams
add 34.217.1 g of wat 97.2223 grams
actual weight of KMnO4 added _____ grams
actual weight of water added _____ grams

)ate prepared: 10/25/99
Prepared by: Richard T Hall
Work Package Number: W53400

1M NaMnO4 97.2792 g
34.2 mL

Project No. _____ Date of Work _____
Entered By Richard T Hall Date 10/25/99
Disclosed To and Understood By
Signed 1

Serene T1-063 9/27/99

Added 1 l of solution Archive #AV-107 to 1000 mL flask.
Capped & stored overnight (last night)
Calibrated balance w/ weights in cell

Weight of Cal Wt	Weight Measured
100g	100.0g
1000g	999.9g
5000g	4999.7g

waste added 1160.8936 grams
− 66.9736
1093.9200 grams

Weight of flask 2062.5 g (w/ rubber stopper)
Beaker (empty) 14.8559 g (before use)

Weight Beaker (empty) 14.8513 g (after use)
Weight 50 mL ~~bottle~~ 15.0983 g (empty) } 66.9736 grams removed
Weight 50 mL bottle 81.9823 g (full)

Weight of flask 1995.9 g (w/ rubber stopper) (67.1 grams)

Adding $Sr(NO_3)_2$ we see white flecks in black solution increased w/ more Sr addition.

Quite a few flecks after completed. As it stirs, the flecks are getting smaller & the solution is lighter br color.

2.1 / 145.4 / 179.7 ingredients added

Added $NaMnO_4$. Solution turned dark brown again. White flecks not seen anymore. Solution is milk-chocolate brow

Weight of flask 2175.1 g (w/ rubber stopper)

Began 4 hr heat at 8:33
Sampled initial from the 50mL pulled before any chemical addi
MR-01 42.9740 g Initial Wt = 16.8154 g

MR-02 Initial Wt = 16.8161 g

Project No. _____ Date of Work _____
Entered By J T Brooks Date 10/7/99
Disclosed To and Understood By:
Signed 1. Edward J. Hollen Date 12/14/99
2. _____ Date _____

12:33 Time for 4 hr digestion finished.
Temp ranged from 47.5 - 53.4 C over 4 hrs.
12:41 T = 52.0 C Stirrer and Heat off
Finished with 4 hr. digestion.
Camera recording.

1:08 46.4 C = T waste
1:26 T = 44.1
2:00 T = 40.4
2:36 T = 37.3
2:51 T = 36.3
3:22 T = 34.5
4:06 T = 32.7
4:18 T = 32.3

4:30 Filtering - The solution is filtering very well There is already 700+ ml of filtrate in the collection bottle. Swirling to add last 1/2 to the * Stir bar slid into the filter. Will retrieve later.
5:00 Filter Cake is mudd like.

~ 6:05pm Mostly completed filtration of treated archive AN-107

10/28/99

Filled 250 mL container w/ treated, filtered archive A

Measured density of treated, filtered archive AN-107

Filled MR-03 43.9364 g - vial w/ lid

Weighed receiver bottle & filter unit.

Weight of big flask + Stopper = 869.3 g

Original tare w/o Stopper = 736 g

Stopper = 85.2 g

Project No. _____ Date of Work _____

Entered By D.L. Brooks Date 10/28/99

Disclosed To and Understood By,

Signed 1. Richard T. Hall Date 12/14/99

2. _____ Date _____

Appendix C: Analytical Data

Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ...
ICPAES Data Report

RT HALLEN
Date 9/3/99
Route
File 71-071
Copy

Project: # 29953
Client: K. Brooks

original

final data report

ACL Number(s): 99-2255 through 99-2260

Client ID: "Mn-23" through "Mn-28"

ASR Number: 5457

Total Samples: 6

Procedure: PNL-ALO-211, "Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry" (ICP-AES).

Analyst: DR Sanders

Analysis Date (Filename): 08-04-99 (A0538)

See Chemical Measurement Center 98620: ICP-325-405-1 File for Calibration and Maintenance Records.

M&TE Number: ICPAES instrument -- WB73520
Mettler AT400 Balance -- Ser.No. 360-06-01-029

Jessy Wagner 8-13-99
Reviewed by

MW Mar 8-17-99
Concur

Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ...
ICPAES Data Report

Six radioactive aqueous samples, Mn-23 through Mn-28 (ACL# 99-2255 through 99-2260), were analyzed by ICPAES after preparation by the Sample Receiving and Preparation Laboratory (SRPL). Samples were prepared by SRPL using PNL-ALO-128 acid digestion procedure. All samples were prepared using plastic labware. Approximately 5ml aliquots of sample solution were weighed, processed and diluted to a final volume of 20ml. All liquid samples were caustic, salt solutions prior to processing. Sample Mn-28 (ACL# 99-2260) was filtered after being diluted to 20 ml using 0.45 um membrane filter.

All results reported are in µg/g including liquid samples as requested by client. All results have been corrected for preparation and analytical dilution. Volumes and weights are recorded on bench sheets (included with raw data, etc.). Analytes of interest include Al, Ba, Ca, Cd, Co, Cr, Cu, Fe, K, La, Mg, Mn, Mo, Na, Ni, Pb, Si, Ti, U, and Zn. No changes have been made relative to the preliminary report provided earlier to the client.

All quality control checks met tolerance requirements for analytes of interest except as noted below. Following is a list of quality control check measurement results relative to ICPAES analysis tolerance requirements under MCS-033.

Five fold serial dilution:
(Aqueous samples) All results were within tolerance limit of ≤ 10% after correcting for dilution.

Duplicate RPD (Relative Percent Difference):
(Aqueous samples) All analytes of interest were recovered within tolerance limit of ≤ 20% relative percent difference (RPD).

Post-Spiked Samples (Group A):
(Aqueous samples) All analytes of interest were recovered within tolerance of 75% to 125%.

Post-Spiked Samples (Group B):
(Aqueous samples) All analytes of interest were recovered within tolerance of 75% to 125%.

Blank Spike:
(Aqueous samples) All analytes of interest in the blank spike were recovered within tolerance limit of 80% to 120%.

Matrix Spiked Sample:
(Aqueous samples) All analytes of interest in the matrix-spike were recovered within tolerance limit of 75% to 125% except for Ba and Pb. Matrix-spike recovery for barium in sample Mn-22 (ACL# 99-2259) was only about 5%. Lead recovery in the same sample was about 41%. Low recovery for the two analytes may be caused by moderate to high concentration of sulfate in the sample. Post-spike and blank-spike recovery for all analytes of interest was recovered within tolerance limit of 75% to 125%.

Quality Control Check Standards:
Concentration of all analytes of interest was within tolerance limit of ± 10% accuracy in the standards: QC_MCVA and QC_MCVB. Calibration Blank (ICP98.0) concentration was less than two times IDL.

High Calibration Standard Check:
Verification of the high-end calibration accuracy for all analytes of interest except lanthanum and uranium was within ± 5% tolerance. The high-end calibration accuracy for lanthanum was somewhat low, about –6.9% and uranium was also low by about –5.7%. This will cause results for lanthanum and uranium concentration to appear lower than what is actually present in the samples. Lanthanum and uranium concentration measurements were all below EQL (and below MRQ).

Process Blank:
(Aqueous samples) All analytes of interest were within tolerance limit of ≤ EQL or < 5% of sample concentration.

Laboratory Control Standard (LCS):
(Aqueous samples) No LCS was prepared for PNL-ALO-128 acid digested samples.

Analytes other than those requested by the client are for information only. Please note bracketed values listed in the data report are within ten times instrument detection limit and have a potential uncertainty much greater than 15%. See attached ICPAES data results.

Comments:

1) "Final Results" have been corrected for all laboratory dilution performed on the sample during processing and analysis unless specifically noted.

2) Detection limits (DL) shown are for acidified water. Detection limits for other matrices may be determined if requested.

3) Routine precision and bias is typically ± 15% or better for samples in dilute, acidified water (e.g. 2% v/v HNO_3 or less) at analyte concentrations greater than ten times detection limit up to the upper calibration level. This also presumes that the total dissolved solids concentration in the sample is less than 5000 µg/mL (0.5 per cent by weight).

4) Absolute precision, bias and detection limits may be determined on each sample if required by the client.

5) The maximum number of significant figures for all ICP measurements is 2.

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

Det. Limit (ug/mL)	Multiplier= ALO#= Client ID= Run Date= (Analyte)	3.2 99-2255-PB @1 Process Blank 8/4/99 ug/g	16.3 99-2255 @5 Mn-23 8/4/99 ug/g	16.4 99-2256 @5 Mn-31 8/4/99 ug/g	16.3 99-2257 @5 Mn-32 8/4/99 ug/g	15.9 99-2258 @5 Mn-21 8/4/99 ug/g
0.015	Ag	–	–	–	–	–
0.060	Al	–	119	105	106	133
0.080	As	–	–	–	–	–
0.050	B	–	13.8	16.4	13.8	18.5
0.010	Ba	–	[0.33]	–	–	[1.3]
0.010	Be	–	–	–	–	–
0.100	Bi	–	–	–	–	–
0.100	Ca	–	130	129	129	227
0.015	Cd	–	23.2	22.6	22.5	27.0
0.100	Ce	–	–	–	–	[9.7]
0.025	Co	–	[1.8]	[1.8]	[1.8]	[2.0]
0.020	Cr	–	35.7	36.9	36.1	58.6
0.015	Cu	[0.13]	11.5	14.1	12.7	12.8
0.050	Dy	–	–	–	–	–
0.100	Eu	–	–	–	–	–
0.025	Fe	[0.13]	8.00	7.56	5.82	445
2.000	K	–	604	584	592	665
0.025	La	–	[0.77]	[0.71]	[0.74]	9.46
0.005	Li	–	[0.34]	[0.26]	[0.25]	[0.25]
0.100	Mg	–	–	–	–	–
0.005	Mn	–	1.13	[0.48]	[0.50]	44.1
0.030	Mo	–	13.2	12.9	12.9	15.0
0.100	Na	[0.75]	84,600	84,200	86,200	104,000
0.100	Nd	–	[2.6]	[2.9]	[2.9]	29.0
0.030	Ni	–	189	184	185	217
0.100	P	–	166	162	163	184
0.060	Pb	–	69.5	66.9	67.4	134
0.300	Pd	–	–	–	[4.9]	[16]
0.300	Rh	–	–	–	–	[4.8]
0.075	Ru	–	13.0	12.5	12.7	14.8
0.050	Sb	–	–	–	–	–
0.050	Se	–	[1.0]	[0.94]	[1.0]	[1.00]
0.100	Si	–	38.2	43.8	34.4	41.9
1.000	Sn	–	–	–	–	–
0.005	Sr	–	115	171	159	1.14
0.500	Te	–	–	–	–	–
0.800	Th	–	–	–	–	–
0.005	Ti	–	–	–	–	0.943
0.250	Tl	–	–	–	–	–
2.000	U	–	[40]	[38]	[41]	[48]
0.015	V	–	–	–	–	–
0.500	W	–	[60]	[59]	[59]	[70]
0.010	Y	–	[0.44]	[0.59]	[0.56]	2.81
0.020	Zn	[0.18]	5.19	5.48	5.39	9.36
0.025	Zr	–	[1.1]	[1.2]	[0.94]	14.3

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.

2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.

3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

Det. Limit (ug/mL)	Multiplier= ALO#= Client ID= Run Date= (Analyte)	16.3 99-2259 @5 Mn-22 8/4/99 ug/g	16.3 99-2259-DUP @5 Mn-22 8/4/99 ug/g	80.5 99-2260 @25 Mn-28 8/4/99 ug/g		
0.015	Ag	--	--	--	--	--
0.060	Al	105	107	121	--	--
0.080	As	--	--	--	--	--
0.050	B	13.1	13.4	[15]	--	--
0.010	Ba	--	--	[1.9]	--	--
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.100	Ca	126	130	220	--	--
0.015	Cd	22.8	23.4	22.6	--	--
0.100	Ce	--	--	[15]	--	--
0.025	Co	[1.7]	[1.8]	--	--	--
0.020	Cr	34.4	35.2	64.2	--	--
0.015	Cu	11.1	11.3	12.1	--	--
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	4.32	4.40	687	--	--
2.000	K	597	614	[600]	--	--
0.025	La	[0.63]	[0.67]	[11]	--	--
0.005	Li	[0.21]	[0.23]	[0.60]	--	--
0.100	Mg	--	--	--	--	--
0.005	Mn	3.73	3.84	2,810	--	--
0.030	Mo	12.8	13.3	[12]	--	--
0.100	Na	90,700	85,100	85,200	--	--
0.100	Nd	[1.9]	[2.0]	[38]	--	--
0.030	Ni	185	191	188	--	--
0.100	P	162	165	138	--	--
0.060	Pb	67.2	69.1	140	--	--
0.300	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
0.075	Ru	12.6	13.0	[13]	--	--
0.050	Sb	--	--	--	--	--
0.050	Se	[0.86]	[1.00]	[4.6]	--	--
0.100	Si	29.5	30.4	[44]	--	--
1.000	Sn	--	--	--	--	--
0.005	Sr	110	114	4,770	--	--
0.500	Te	--	--	--	--	--
0.800	Th	--	--	--	--	--
0.005	Ti	--	--	[1.3]	--	--
0.250	Tl	--	--	--	--	--
2.000	U	[38]	[39]	--	--	--
0.015	V	--	--	--	--	--
0.500	W	[59]	[60]	[47]	--	--
0.010	Y	[0.32]	[0.31]	[3.6]	--	--
0.020	Zn	5.29	5.74	[8.4]	--	--
0.025	Zr	[0.83]	[0.72]	--	--	--

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.

2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.

3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Radioanalytical Applications Team

ASR # 5457

WP# W51304

File: L:\radchem\hydroxide\asr5457
Analysis Date: 8/3/99
Print Date: 8/4/99

Hydroxide and Alkalinity Determination
Governing Procedures: **PNL-ALO-228:** Determination of Hydroxyl (OH-) and Alkalinity of Aqueous Solutions, Leachates and Supernates and Operation of Brinkman 636 Auto-Titrator
Equip # **WB76843**
Lab Loc. 525

Analyst: 8/4/99
Reviewer: 8/4/99

Titrant	Molarity
HCl	**0.2034**

Std. & Spike	Molarity
NaOH	**0.1018**

RPG #	Sample ID		Sample Vol. (mL)	Sample Wt. (g)	Density g/mL	Titrator Routine #	Initial pH reading	OH 1st Equivalence Point Titrant Vol. (mL)	pH	Found millimoles base	Molarity base	millimole RPD
99-2258	**Mn-21**		0.100	0.1271	1.271	1	11.558	0.423	10.624	0.086	0.86	
99-2258	**Mn-21**	Replicate	0.200	0.2547	1.274	2	11.759	0.855	10.750	0.174	0.87	1.06%
99-2259	**Mn-22**		0.200	0.2485	1.243	3	11.702	0.723	10.67	0.147	0.74	
99-2259	**Mn-22**	Replicate	0.200	0.2494	1.247	4	11.707	0.724	10.699	0.147	0.74	0.14%
QC Data:												
Reag. Blk.			5.00			1	6.143				**OH % Recovery, Acc**	
Standard 1	0.1018 N NaOH		5.000	5.0036	1.001	2	12.026	2.492	7.918	0.5069	**99.6%**	Std 1
Standard 2	0.1018 N NaOH		5.000	5.0196	1.004	3	11.89	2.486	7.578	0.5057	**99.3%**	Std 2
99-2258MS	Mn-21 + 2mL 0.1N NaOH		0.100	0.1256	1.256	5	11.846	1.338	10.611	0.272	**91.2%**	MS
99-2259MS	Mn-22 + 2mL 0.1N NaOH		0.100	0.1229	1.229	6	11.702	1.292	10.640	0.263	**92.9%**	MS

Buffer	VWR Lot #	CMS#	Expire Date
10	981659-24	144109	Jul-00
4	981583-24	144107	Jun-00
7	981894-24	144108	Aug-00

Performance checks

Balance # 360--01-06-037

		Vol.	Wt.
Pipet #	H30762	5.00	4.944
Pipet #	2734494	0.500	0.496
Pipet #	120737	0.100	0.1013
Pipet #	120737	0.200	0.1997

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Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Radioanalytical Applications Team

ASR # 5457
WP# W51304

File: L:\radchem\hydroxide\asr5457
Analysis Date: 8/3/99
Print Date: 8/4/99

Hydroxide and Alkalinity Determination
Governing Procedures: PNL-ALO-228: Determination of Hydroxyl (OH-) and Alkalinity of Aqueous Solutions, Leachates and Supernates
and Operation of Brinkman 636 Auto-Titrator
Equip # WB76843
Lab Loc. 525

Analyst: 8/4/99
Reviewer: 8/4/99

Titrant	Molarity
HCl	0.2034

RPG #	Sample ID		Sample Vol. (mL)	CO3 2nd Equivalence Point Titrant Vol. (mL)	pH	Found millimoles base	Molarity base	millimole RPD	HCO3 3rd Equivalence Point Titrant Vol. (mL)	pH	Found millimoles base	Molarity base	millimole RPD
99-2258	Mn-21	0	0.100	0.859	7.658	0.089	0.887		1.314	4.427	0.093	0.93	
99-2258	Mn-22	Replicate	0.200	1.720	7.793	0.176	0.880	0.81%	2.617	4.526	0.182	0.91	1.4%
99-2259	Mn-22	0	0.200	1.460	7.670	0.150	0.750		2.210	4.573	0.153	0.76	
99-2259	Mn-22	Replicate	0.200	1.454	7.808	0.148	0.742	0.95%	2.211	4.581	0.154	0.77	0.9%
racy							CO3 % Recovered						
Standard 1	0.1018 N NaOH		5.000	2.559	4.054	0.01363	0.003	sample					
Standard 2	0.1018 N NaOH		5.000	2.566	3.879	0.01627	0.003	sample			HCO3 % recovered		
99-2258MS	Mn-21 + 2mL 0.1N NaOH		0.100	1.825	7.723	0.09906	112.1%	sample	2.324	4.573	0.1015	110.5%	sample
99-2259MS	Mn-22 + 2mL 0.1N NaOH		0.100	1.715	7.7	0.08604	115.3%	sample	2.146	4.495	0.0877	114.4%	sample

Matrix spike recovery is calculated as follows:
Spike = 2.00 mL 0.1018 N NaOH was added to the 0..100-mL of sample for each matrix spilke.
SpikeTitrant vol. (sample @ .1mL + spike) - SampleTitrant vol. (average sample only equated to .1mL) * 0.2034 N (HCl titrant) = meq. OH
meq OH / 2.00 mL added = meq OH/mL found / 0.1018 N OH added * 100 = % recovered.

Prep record on 0.2034 M HCl is on following page.

Chem Rec_51a

Prep date: 4/18/99

Preparation of Standardized 0.2 M HCl

WP# K51300

Standardized O.1021 M NaOH will be re-checked and then used to standardized the ~ 0.1 M HCl solution. The 0.1021 M NaOH was prepared in Chem Rec_37 (see Chem Rec_37 --prep.date 2-25-98 for original data) and re-verified against NIST SRM84j Potassium Acid Phthalate KHC8H404 (KAP) = 204.23 g/mole -- Barcode # 52232 --- (see below verification check).
The re-standardized value of 0.1018 M NaOH was reassigned to this NaOH solution with a revised Expiration Date of Feb. 2000.
Prepared 1- liters of ~0.2 M HCl by diluting 100 mL of 1.029M HCl (Chemrec_10) to 0.5 L with DI. H2O.
20 mL aliquots of 0.2 M HCl were were neutralized to the phenopthalien endpoint using the re-standardized 0.1018 M NaOH. The volume of NaOH is accurate to +/- 0.02mL and the pipitting error is estimated to be < 1% @ 1s. Thus total error is < 3 % for the measurements

NaOH Molarity veification

Verification Test #	Wt. of KAP	Vol. of **0.1021M NaOH** to neutralize	NaOH Molarity =a * 1000 / b * 204.23	Molarity Error +/- @ 1 s
1	0.80894	38.95	0.1017	
2	0.80582	38.84	0.1016	
3	0.96233	46.12	0.1022	
		Ave=	**0.1018**	0.0003
			re-certified value	

Titration Id.	aliquot of sample	Vol. of **0.1018M NaOH** to neutralize	**Molarity of Acid** in Sample	Molarity Error +/- @ 1 s
1	20.00	39.88	0.2030	
2	20.00	39.92	0.2032	
3	20.00	40.04	0.2038	
		Ave Molarity HCl =	**0.2034**	0.00042

8/4/99

Analyst/Date

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Radioanalytical Applications Team

99-2255
8/6/99

Client : Brooks

Cognizant Scientist: T R Greenwood Date : 8-6-99

Concur : C Soderquist Date : 8-6-99

Sr-90 (PNL-ALO-476, 431)
Gamma (PNL-ALO-450)

Measured Activities (uCi/ml)

ALO ID Client ID	Co-60 Error %	Eu-152 Error %	Cs-137 Error %	Eu-154 Error %	Sb-125 Error %	SnSb-126 Error %	Eu-155 Error %	Am-241 Error %
99-2255PB Process Blank	<2.E-5	<4.E-5	<2.E-5	<4.E-5	<3.E-5	<1.E-5	<5.E-5	<9.E-5
99-2255 Mn-23	6.21E-2 2%	<2.E-3	6.52E-2 2%	2.20E-2 2%	<9.E-4	4.31E-4 22%	1.65E-2 3%	9.48E-3 8%
99-2256 Mn-31	6.02E-2 2%	<2.E-3	4.03E-2 2%	2.95E-2 2%	1.12E-3 25%	3.89E-4 16%	2.15E-2 3%	9.77E-3 8%
99-2257 Mn-32	6.22E-2 2%	<2.E-3	4.45E-2 2%	2.86E-2 2%	<9.E-4	2.69E-4 17%	2.11E-2 3%	1.09E-2 7%
99-2258 Mn-21	7.74E-2 2%	5.02E-3 8%	2.83E-2 2%	2.91E-1 2%	<2.E-3	<6.E-4	2.08E-1 3%	2.19E-1 5%
99-2259 Mn-22	6.68E-2 2%	<2.E-3	2.31E-2 2%	1.65E-2 2%	<8.E-4	3.89E-4 16%	1.19E-2 4%	5.56E-3 15%
99-2259 Dup 1 Mn-22	6.68E-2 2%	<2.E-3	2.34E-2 2%	1.73E-2 2%	<8.E-4	3.86E-4 18%	1.19E-2 4%	7.27E-3 15%
RPD	0%		1%	5%		1%	0%	27%
99-2259 Dup 2 Mn-22	6.55E-2 2%	<2.E-3	2.20E-2 2%	1.64E-2 2%	<8.E-4	2.87E-4 21%	1.21E-2 5%	5.04E-3 17%
99-2260 Mn-28	6.38E-2 2%	6.14E-3 9%	3.05E-2 3%	3.59E-1 1%	<2.E-3	<6.E-4	2.59E-1 3%	2.63E-1 11%

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Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Radioanalytical Applications Team

99-2255
8/6/99

Client : Brooks

Cognizant Scientist: [signature] Date : 8-6-99

Concur : [signature] Date : 8-6-99

Sr-90 (PNL-ALO-476, 431)
Gamma (PNL-ALO-450)

Measured Activities (uCi/g)

ALO ID Client ID	Co-60 Error %	Eu-152 Error %	Cs-137 Error %	Eu-154 Error %	Sb-125 Error %	SnSb-126 Error %	Eu-155 Error %	Am-241 Error %
99-2255PB Process Blank	<2.E-5	<4.E-5	<2.E-5	<4.E-5	<3.E-5	<1.E-5	<5.E-5	<9.E-5
99-2255 Mn-23	5.09E-2 2%	1.64E-3	5.34E-2 2%	1.80E-2 2%	7.37E-4	3.53E-4 22%	1.35E-2 3%	7.77E-3 8%
99-2256 Mn-31	4.94E-2 2%	1.64E-3	3.30E-2 2%	2.42E-2 2%	9.18E-4 25%	3.19E-4 16%	1.76E-2 3%	8.01E-3 8%
99-2257 Mn-32	5.09E-2 2%	1.64E-3	3.64E-2 2%	2.34E-2 2%	7.37E-4	2.20E-4 17%	1.73E-2 3%	8.92E-3 7%
99-2258 Mn-21	6.16E-2 2%	4.00E-3 8%	2.25E-2 2%	2.32E-1 2%	1.59E-3	4.78E-4	1.66E-1 3%	1.74E-1 5%
99-2259 Mn-22	5.45E-2 2%	1.63E-3	1.88E-2 2%	1.35E-2 2%	6.52E-4	3.17E-4 16%	9.70E-3 4%	4.53E-3 15%
99-2259 Dup 1 Mn-22	5.44E-2 2%	1.63E-3	1.91E-2 2%	1.41E-2 2%	6.52E-4	3.15E-4 18%	9.70E-3 4%	5.92E-3 15%
RPD	0%		1%	5%		1%	0%	27%
99-2259 Dup 2 Mn-22	5.33E-2 2%	1.63E-3	1.79E-2 2%	1.34E-2 2%	6.51E-4	2.34E-4 21%	9.85E-3 5%	4.10E-3 17%
99-2260 Mn-28	5.15E-2 2%	4.95E-3 9%	2.46E-2 3%	2.90E-1 1%	1.61E-3	4.84E-4	2.09E-1 3%	2.12E-1 11%

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Radioanalytical Applications Team

99-2255
8/6/99

Client : Brooks

Cognizant Scientist: C. Soderquist Date : 8-6-99

Concur : L. R. Greenwood Date : 8-6-99

Sr-90 (PNL-ALO-476, 431)
Gamma (PNL-ALO-450)

ALO ID Client ID	Sr-90 μCi/mL ± 1s	Sample Density, g per mL	Sr-90 μCi/g ± 1s
99-2255PB Process Blank	<5.E-3	1	<5.E-3
99-2255 Mn-23	1.96E+0 ± 4%	1.221	1.61E+0 ± 4%
99-2255DUP Mn-23	2.22E+0 ± 4%	1.221	1.82E+0 ± 4%
RPD	12%		
99-2256 Mn-31	1.99E+0 ± 4%	1.220	1.63E+0 ± 4%
99-2257 Mn-32	2.22E+0 ± 4%	1.221	1.82E+0 ± 4%
99-2258 Mn-21	4.84E+1 ± 4%	1.256	3.85E+1 ± 4%
99-2259 Mn-22	1.87E+0 ± 3%	1.228	1.52E+0 ± 3%
99-2260 Mn-28	3.31E+1 ± 3%	1.240	2.67E+1 ± 3%
Matrix Spike	88%		
Reagent Spike	102%		
Blank	<5.E-3		

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Radioanalytical Applications Team

99-2255
8/6/99

Client : Brooks

Cognizant Scientist: C Sodergren Date : 8-6-99

Concur : L P Greenwood Date : 8/6/99

Sr-90 (PNL-ALO-476, 431)
Gamma (PNL-ALO-450)

	Densities of Samples				
ALO ID / Client ID	Gross	Tare	Net	Volume	Density
99-2255 Mn-23	14.1799 g	8.0603 g	6.1196 g	5.01 mL	1.221 g/mL
99-2256 Mn-31	14.3510 g	8.2358 g	6.1152 g	5.01 mL	1.220 g/mL
99-2257 Mn-32	14.1705 g	8.0474 g	6.1231 g	5.01 mL	1.221 g/mL
99-2258 Mn-21	14.4554 g	8.1599 g	6.2955 g	5.01 mL	1.256 g/mL
99-2259 Mn-22	14.2015 g	8.0536 g	6.1479 g	5.01 mL	1.226 g/mL
99-2259 Dup 1 Mn-22	14.3323 g	8.1806 g	6.1517 g	5.01 mL	1.227 g/mL
99-2259 Dup 2 Mn-22	14.2551 g	8.0797 g	6.1754 g	5.01 mL	1.232 g/mL
				Average, n = 3	1.228 g/mL
				±	0.2%
99-2260 Mn-28	14.3292 g	8.1147 g	6.2145 g	5.01 mL	1.240 g/mL

Volume is known to ± 0.2%, 1s.

Analytical Chemistry Laboratory (ACL) Analytical Services Request (ASR)

(Cover Page ... information applicable to all samples in series)

Requested By: Kristen Brooks (Print Name) | (Signature/Date) 9/3/99 | 6-2233 (Phone) | K6-24 (MSIN)

Requester - Please Complete All Fields In This Section, Unless Specified "Optional" or ASR is a Revision

Request ID (optional): _____

PNL Project Number (if known): 29953

Work Order/Pkg.: W53400

Cost Estimate ($): _____

Protocol Requirement: X None __RCRA __CERCLA, or

Other (specify): _____

Hold Time Requirement: X None __RCRA __CERCLA, or

Other (specify): _____

TPA Support: X No, or

Milestone No.: _____

QA Plan: __MCS-033, or

Other ACL QA Plan (specify): SBMS

Additional QA Requirements: X No, or

Reference Doc.: _____

ACL COC Req'd (PNL-ALO-010): X No __Yes

Sample Storage Requirements: X No __Refrigerate, or

Other (specify): _____

Date Sampled (optional): _____

Time Sampled (optional): _____

Matrix: __Samples vary (specify on Request Page), or

Liquid: X Aqueous __Organic __Multi-phasic

Solid: __Soil __Sludge __Sediment __Glass

__Filter __Smear __Metal __Organic __Other Solids

Solid/Liquid Mixture: __ Gas: __

Biological: __Tissue __Urine __Feces

Process Knowledge: __Sample Information Check List,

or Reference Doc.: _____

PCBs Present: X No __Yes

Sample Disposition ...

Untreated Sample(s): __Return X Dispose __Store, or

Reference Doc.: _____

Prep'd Sample(s): X Dispose __Return __Store, or

Reference Doc.: _____

Additional Instructions: __No, or

Reference Doc.: _____

Date Report Req'd: 12-1-99

Send Report to: Rich Hallen

MSIN: K2-12 Phone: 375-6919

Fax (optional): 372-4732

For ACL Use Only ... Do Not Complete This Section

Date Delivered: 11/1/99

Time Delivered (optional): _____

Deliv. By (if known): Mike Mann

Received By: FU Hoopes

Resp. ACL Mgr.: _____

Signature/Date: _____

Job Group (optional): _____

Sample Group (optional): _____

PNL Impact Level: __1 __2 __3

DQ Review Req'd: X No __Yes ACL Waste: X No __Yes

ASR Number: _____ Revision: __Yes

ACL Numbers: _____

Analytical Chemistry Laboratory (ACL) Analytical Services Request (ASR)
(Request Page ... information specific to individual samples)

ACL Number (for ACL use only)	Client Sample Number (list each sample separately)	Sample Description	Analyses Requested	Test Name (for ACL use only)	Library (for ACL use only)	
	MR-01	Unfiltered Supernate	Acid digest, GEA, Sr-90, ICP(Na)			
	MR-02	Filtered Supernate	Tc-99 (β count with out oxidation)			
	MR-03	Treated & filtered supernate				

ASR Number (for ACL use only): _____

Page ___ of ___

Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ...
ICPAES Data Report

Project: 29953
Client: K.P. Brooks

ACL Number(s): 00-0292 through 00-0294

Client ID: "MR-01" through "MR-03"

ASR Number: 5570

Total Samples: 3

Procedure: PNL-ALO-211, "Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry" (ICP-AES).

Analyst: D.R. Sanders

Analysis Date (Filename): 11-12-99 (A0552)

See Chemical Measurement Center 98620: ICP-325-405-1 File for Calibration and Maintenance Records.

M&TE Number: ICPAES instrument -- WB73520
Mettler AT400 Balance -- Ser.No. 360-06-01-029

Jerry Wagner 1-17-00
Reviewed by

MW Weir 1-18-00
Concur

Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ...
ICPAES Data Report

Three radioactive aqueous samples, **MR-01** through **MR-03** (ACL# 00-0292 through 00-0294) were analyzed by ICPAES after samples were processed by the Shielded Analytical Laboratory (SAL). ALO-128 acid digestion procedure was used to process the three samples and a process blank. Approximately 5 to 6 ml sample aliquots were processed and diluted to a corrected final volume of approximately 23 to 24 ml. Sodium was the only analyte requested. Analytical dilutions of 5-fold to 50-fold were necessary due to the high concentration of sodium present in the sample.

Measurement results have been corrected for preparation and analytical dilution. Sample results are reported as µg/g as requested. Weights and volumes used have been recorded on bench sheets and are included with this report. Analytes reported other than sodium are for information only.

Quality control check-standard results for sodium met tolerance requirements except as noted below. Following is a list of quality control measurement results relative to ICPAES analysis tolerance requirements under MCS-033.

Five fold serial dilution:
(Aqueous sample) All results for sodium in the diluted sample is within tolerance limit of ≤ 10% after correcting for dilution.

Duplicate RPD (Relative Percent Difference):
(Aqueous sample) Sodium is recovered within tolerance limit of ≤ 20% relative percent difference (RPD).

Post-Spiked Samples (Group A):
(Aqueous sample) Sodium was recovered within tolerance of 75% to 125%.

Blank Spike:
(Aqueous sample) None.

Matrix Spiked Sample:
(Aqueous sample) None.

Quality Control Check Standards (aqueous sample):
Concentration for Sodium is within tolerance limit of ± 10% accuracy for QC_MCVA, ICP98.0 and QC_SSTMCV check standards.

High Calibration Standard Check (aqueous sample):

Verification of the high-end calibration concentration for sodium is within tolerance of ± 5% accuracy.

Process Blank:

(Aqueous sample) Sodium was detected in the process blank but at a concentration less than 5% of the concentration measured in any of the samples and is within tolerance limits.

Laboratory Control Standard (LCS):

(Aqueous sample) None prepared for ALO-128 acid digestion procedure.

Please note bracketed values listed in the data report are within ten times instrument detection limit and have a potential uncertainty much greater than 15%.

Comments:

1) "Final Results" have been corrected for all laboratory dilution performed on the sample during processing and analysis unless specifically noted.

2) Detection limits (DL) shown are for acidified water. Detection limits for other matrices may be determined if requested.

3) Routine precision and bias is typically ± 15% or better for samples in dilute, acidified water (e.g. 2% v/v HNO_3 or less) at analyte concentrations greater than ten times detection limit up to the upper calibration level. This also presumes that the total dissolved solids concentration in the sample is less than 5000 µg/mL (0.5 per cent by weight).

4) Absolute precision, bias and detection limits may be determined on each sample if required by the client.

5) The maximum number of significant figures for all ICP measurements is 2.

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

Det. Limit (ug/mL)	Multiplier= ALO#= Client ID= Run Date= (Analyte)	4.8 00-0292-PB Process Blank 11/12/99 ug/g	19.1 00-0292 @5 MR-01 11/12/99 ug/g	19.0 00-0292-DUP @5 MR-01 11/12/99 ug/g	19.1 00-0293 @5 MR-02 11/12/99 ug/g	18.4 00-0294 @5 MR-03 11/12/99 ug/g
0.025	Ag	--	--	--	--	--
0.060	Al	5.54	156	154	141	117
0.250	As	--	--	--	--	--
0.050	B	14.0	18.9	17.4	19.3	18.4
0.010	Ba	--	2.08	2.07	[0.64]	--
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	--	248	244	254	144
0.015	Cd	--	29.1	28.9	30.0	25.0
0.200	Ce	--	[15]	[14]	[6.1]	--
0.050	Co	--	[2.1]	[2.1]	[2.2]	[1.9]
0.020	Cr	--	71.8	71.0	50.4	31.8
0.025	Cu	--	13.1	13.0	13.8	11.5
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	[0.27]	690	684	219	11.7
2.000	K	--	724	723	767	659
0.050	La	--	14.1	14.1	12.5	--
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	--	139	138	24.6	27.4
0.050	Mo	--	15.7	15.5	16.3	13.7
0.150	Na	26.5	91,500	93,900	96,000	104,000
0.100	Nd	--	42.4	42.1	31.8	[2.9]
0.030	Ni	[0.27]	235	233	241	203
0.100	P	--	201	197	214	182
0.100	Pb	--	165	162	144	81.2
0.750	Pd	--	[22]	[21]	[15]	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	28.0	[23]	[23]	[19]	[35]
1.500	Sn	--	--	--	--	--
0.015	Sr	--	[1.2]	[1.2]	[1.2]	109
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	[2.2]	[2.1]	[0.57]	--
0.500	Tl	--	--	--	--	--
2.000	U	--	[53]	[45]	[53]	[47]
0.050	V	--	--	--	--	--
2.000	W	--	[78]	[78]	[79]	[66]
0.050	Y	--	[3.7]	[3.7]	[3.0]	--
0.050	Zn	--	[8.8]	[8.7]	[6.5]	[4.7]
0.050	Zr	--	20.2	16.6	15.4	[2.9]

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.

2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.

3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

Pacific Northwest National Laboratory
Radiochemical Processing Laboratory
Shielded Facility Operation Team

Corrected Copy
11/5/99

SAMPLE PREP SHEET
(325 SHIELDED ANALYTICAL LABORATORY)

TI/ARF NO.: ASR5570 PROJECT NO.: 29553 WBS NO.: _____ SAMPLE TYPE: AQUEOUS

ISSUED BY: RT STEELE DATE: 11/01/99 PREP TYPE: ACID DIGEST (ALO-128)

ANALYST: [signature] DATE: 11-3-99 CHAIN OF CUSTODY RQD: NONE

REVIEW: Rick T. Steele DATE: 11-4-99 QA PLAN: SBMS IMPACT LEVEL: _____

CLIENT: RT HALLEN (K Brooks) CORE ID: N/A TANK ID: AN107

WORK PACKAGE NUMBER	ALO NUMBER	SAMPLE IDENTIFICATION	ANALYTE OR ANALYSIS	SAMPLE WT	Sp.G.[A] (g/mL)	WATER WT (g)	TOTAL VOL (mL)	SPIKE ID	SPIKE VOL (mL)	DILUTION FACTOR	DILUTION MATRIX	PIPET CALIB (mL)	MISC
W45525	00-00292-PB	PROCESS BLANK	1,2	4.9827			~~25.56~~	23.79					
	00-00292	MR-01	1,2	6.2722			~~28.63~~	23.94	RAS 11/5/99				
	00-00292-DUP	MR-01	1,2	6.1909			~~28.29~~	23.48					
	00-00293	MR-02	1,2	6.2789			~~28.73~~	23.98					
	00-00293	MR-03	1,2	6.3154			27.94	23.24				C	

11-15-99

(1) REQUIRED ANALYSES:
ICP-AES (Na)

(2) REQUIRED ANALYSES:
GEA, Sr-90, ~~Tc-99 BY BETA COUNTING WITHOUT OXIDATION~~

(A) Specific Gravity of ORIGINAL material. SEE ASR5570 TABLE 3 FOR ANALYTE LIST AND MRQs

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory

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EXHIBIT I
PAGE 1 of 1

PNL-ALO-128 Nitric and Hydrochloric Acid Extraction of Liquids Using a Dry-Block Heater

Client name: KP Brooks
Work Auth. Doc (WAD): ASR 5570
Tank/Core/Project: Tank AN107
Special instructions

Work package number: W53400
Project number: 29553
PNL QA plan: SBMS
PNL impact level:
Prep. lab (SAL/SRPL/other): SAL
Preparation batch number:

	ACL Sample ID	Client sample ID	Gross Weight (g)	Tare Weight (g)	Net Sample Weight (g)	Sample Volume (ml)	Final Soln Gross Wgt (g)	Final Soln 1mL Wgt (g)	Final Volume (ml)
1	00-00292-PB	PROCESS BLANK	33.7110	28.7283	4.9827 (D.W.)	4.9938	53.3873	1.0268	~~25.5623.7~~ 1
2	00-00292	MR-01	35.1221	28.8499	6.2722	↓	55.6288	1.0832	~~28.6323.9~~ 4
3	00-00292-DUP	MR-01	34.7895	28.5986	6.1909		54.3750	1.0873	~~28.2923.~~ 48
4	00-00293	MR-02	35.2657	28.9868	6.2789		55.2368	1.0844	~~28.7323.~~ 98
5	00-00294	MR-03	35.1118	28.7964	6.3154		54.2790	1.0881 (6)	~~27.94~~ 23. 2
6									
7					density				RTS 11/5/99
8				MR-01	1.256				
9				MR-01 Dup	1.2397				
10				MR-02	1.2573				
11				MR-03	1.2646				
12									
13									
14									

Analyst's sample preparation comments:

5 ml P-pet 23°C
4.9714 $\bar{x}$ = 4.9815
4.9862 S = .0077
4.9767 RSD = .15%
4.9822 Del = 4.9938 ml
4.9908

1 ml P-pet: 23°C
.9873 $\bar{x}$ = .9882
.9889 S = .0011
.9880 RSD = .11%
.9870 Del = .9906 ml
.9897

Spike source:
PNL spike ID number:
Anal. balance M&TE: 360-06-01-016
Sample filtered (yes/no): NO

Other sample preparation worksheets may be substituted at the discretion of the Cognizant Scientist. Use one worksheet per client.

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Radioanalytical Applications Team

00-0292
2/8/00

Client : Brooks

Cognizant Scientist: _____ Date : _____

Concur : _____ Date : _____

Measured Activities (uCi/g)

ALO ID Client ID	Sr-90 Error %
00-0292 MR-01	3.82E+1 3%
00-0292DUP MR-01	2.87E+1 3%
RPD	28%
00-0293 MR-02	4.01E+1 3%
00-0294 MR-03	9.97E-1 3%
Reagent Spike	106%
Blank Spike	99%
Blank	7.69E-3 3%

RT HALLEN
Date 2/8/2000
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Copy record

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Radioanalytical Applications Team

00-0292GEA
2/8/00

Client : Brooks

Cognizant Scientist: _____ Date : _____

Concur : _____ Date : _____

Measured Activities (uCi/g)

ALO ID Client ID	Co-60 Error %	Cs-137 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %
00-0292PB Process Blank	<3.E-5	3.52E-4 6%	<7.E-5	<7.E-5	<9.E-5
00-0292 MR-01	6.04E-2 4%	2.06E-2 13%	2.88E-1 2%	1.93E-1 6%	2.47E-1 11%
00-0292DUP MR-01	6.33E-2 4%	2.53E-2 11%	2.77E-1 2%	1.94E-1 5%	2.72E-1 11%
RPD	5%	20%	4%	1%	10%
00-0293 MR-02	6.27E-2 3%	2.06E-2 8%	1.73E-1 2%	1.21E-1 5%	1.49E-1 11%
00-0294 MR-03	5.16E-2 4%	2.08E-2 10%	2.15E-2 9%	1.51E-2 17%	1.31E-2 25%

RT HALLEN
Date 2/8/2000
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Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Radioanalytical Applications Team

00-0292
2/8/00

Client : Brooks

Cognizant Scientist: _____ Date : _____

Concur : _____ Date : _____

Measured Activities (uCi/g)

ALO ID Client ID	Total Beta Error %
00-0292PB Process Blank	1.86E-2 3%
00-0292 MR-01	8.86E+1 3%
00-0292DUP MR-01	8.84E+1 3%
RPD	0%
00-0293 MR-02	8.10E+1 3%
00-0294 MR-03	2.12E+0 3%
Reagent Spike	124%

RT HALLEN
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Appendix D: Staff and Role/Responsibility

Staff Member	Role/Responsibility
Richard Hallen	Scientist/Technical Leader - Sr/TRU Precipitation
Kriston Brooks	Engineer/CUF System, Entrained Solids Removal, Sr/TRU Precipitation, and Precipitate Removal
Lynette Jagoda	Engineer Associate/CUF System, Entrained Solids Removal, Sr/TRU Precipitation, and Precipitate Removal
Gita Golcar	Scientist/Particle Size Analyses
Don Rinehart	Technician/Hot Cell Tests-Sr/TRU PPT/CUF Operation
Ralph Lettau	Technician/Hot Cell Tests-Sr/TRU PPT/CUF Operation
Dave Ortiz	Technician/Hot Cell CUF Operation and Cleaning
Vaughn Hoopes	Technician/Hot Cell sample prep.
Mac Zumhoff	Technician/Hot Cell Operations

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